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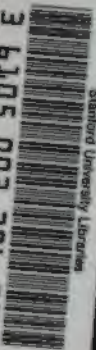
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JOURNAL  
OF THE  
AMERICAN CHEMICAL SOCIETY.

VOLUME III.

*PUBLICATION COMMITTEE:*

ARNO BEHR,  
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FOR THE YEAR 1881.

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# THE AMERICAN CHEMICAL SOCIETY.

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## I.—PROCEEDINGS.

### *Regular Meeting, January 3d, 1881.*

At 9 P.M. the Society was called to order by its President, Prof. CHARLES F. CHANDLER, who occupied the chair. Sixteen members were present.

The minutes of the last meeting were read by Mr. ARTHUR H. ELLIOTT, late Recording Secretary, and adopted.

A copy of the last annual report, as filed with the County Clerk, was read by Mr. ARTHUR H. ELLIOTT, also a report of the last meeting of the Board of Directors, December 13th, 1880.

The following gentlemen were nominated :

As associate member :

Mr. E. K. DUNHAM, School of Mines, N. Y.

Nominated by { ARTHUR H. ELLIOTT,  
C. F. CHANDLER,  
E. WALLER.

As members :

Mr. JAMES F. SLADE, 5 East Thirty-eighth street, N. Y.

Nominated by { JAMES H. STEBBINS, Jr.,  
ARTHUR H. ELLIOTT,  
C. F. CHANDLER.

Mr. THEODORE M. HOPKE.

Nominated by { ARTHUR H. ELLIOTT,  
C. F. CHANDLER,  
M. BENJAMIN.

The following gentlemen resigned their membership :

Prof. IRA REMSEN, resignation accepted unanimously.

Prof. SADDLER, resignation accepted unanimously.

Mr. C. W. DREW, Burlington, Vt., resignation accepted unanimously.

Mr. HARKLEY, resignation accepted unanimously.



It was announced that the Board of Directors had reduced the annual dues to \$5.00.

After considerable discussion their action was adopted with but one dissenting voice.

It was announced that the Board of Directors had altered the day of meeting from Thursday to Monday. After some discussion their action was unanimously adopted.

On motion the Society adjourned.

ALBERT H. GALLATIN,  
*Recording Secretary.*

# THE AMERICAN CHEMICAL SOCIETY.

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## II.—PROCEEDINGS.

*Regular Meeting, February 11th, 1881.*

At 8:30 a quorum being present, but the President and Secretary being absent, the meeting was called to order by Vice-President Leeds.

The chairman stated that in the absence of the Secretary and the minutes, the regular order of business would be suspended, but that some nominations for membership which had been favorably reported upon by the Committee on Nominations, and which had been delayed, should now be acted upon. The names being duly read were as follows:

Prof. E. N. VENABLE, Chapel Hill, North Carolina.

Nominated by { J. W. MALLET,  
C. F. CHANDLER,  
A. H. ELLIOTT.

E. K. DUNHAM, School of Mines, N. Y.

Nominated by { A. H. ELLIOTT,  
C. F. CHANDLER,  
E. WALLER.

Dr. N. GERBER, 45 S. Washington square, N Y.

Nominated by { H. ENDEMANN,  
A. BEHR,  
J. H. STEBBINS, Jr.

JAMES F. SLADE, 5 East Thirty-eighth street, N. Y.

Nominated by { JAS. H. STEBBINS, Jr.,  
A. H. ELLIOTT,  
C. F. CHANDLER.

THEODORE M. HOPKE.

Nominated by { A. H. ELLIOTT,  
C. F. CHANDLER,  
M. BENJAMIN.

On motion, the Vice-President was directed to deposit an affirmative vote for the names unless some objection was made. Such ballot was duly made, and the nominees were all declared elected to membership.

Prof. Leeds then read the papers announced for the evening :

1. "Upon the Invariable Production, not only of Ozone and Hydrogen Peroxide, but also of Ammonium Nitrate, in the Ozonation of Purified Air by Moist Phosphorus." By Dr. A. R. LEEDS.

2. "Upon the Action of Ozone, Oxygen and Nascent Oxygen upon Benzene." By Dr. A. R. LEEDS.

3. "On a New Class of Aromatic Sulphurous Acids." By Dr. A. R. LEEDS.\*

These papers passed to the Committee on Publication without discussion.

Mr. STEBBINS then presented a short paper, "On a New Class of Azo Compounds, properly called 'Tetrazo Compounds.'"

Which also passed to the Committee on Publication without discussion.

Then by invitation from the Secretary, Mr. WALLACE GOULD LEVISON presented a note "On Absorption Spectra of Selenite, Mica, and other Substances Viewed by Polarized Light."

In discussing the paper, Mr. GIUR thought some of the phenomena described might be due to the interspaces of the substances from their foliated structure and general heterogeneity.

Prof. LEEDS also discussed some of the points taken in the paper. Notice was then given that Mr. BENJAMIN, at the next Conversation, would exhibit specimens of Balmain's luminous paints.

The Society then adjourned.

E. R. SQUIBB, VICE-PRES.,

*Acting Secretary.*

---

\* For the present withdrawn by the author.—ED.

III.—UPON THE INVARIABLE PRODUCTION, NOT ONLY OF OZONE AND HYDROGEN PEROXIDE, BUT ALSO OF AMMONIUM NITRATE, IN THE OZONATION OF PURIFIED AIR BY MOIST PHOSPHORUS.

BY DR. A. R. LEEDS.

• As the result of very numerous quantitative experiments performed two years ago, and published in the JOURNAL AMERICAN CHEMICAL SOCIETY, I, 145, and *Chemical News* for August, 1879 ("Upon ammonium nitrite, and upon the by-products obtained in the ozonation of air by moist phosphorus"), I gave the proofs of the statement contained in the heading of this article, and the determination of the amounts of the three substances formed. Forthwith, under the title "Is ozone produced during the atmospheric oxidation of phosphorus?" (*Chemical News*, 40, 96) Mr. Kingzett, without waiting to disprove any of the above propositions by experiment, asserted that "*There is no known process of slow oxidation which has been established to produce ozone,*" and that the gaseous body which I had obtained and experimented upon, was altogether peroxide of hydrogen. Mr. Kingzett insisted that the long series of wash-bottles and purifiers, which I had used to absorb and hold back as far as possible the hydrogen peroxide, did not militate at all against his assumption, for the reason that peroxide of hydrogen suspended in a vesicular condition, might pass through all the washing liquids and constitute the only active agent in the escaping gas. How greatly Mr. Kingzett's views have altered may be seen from the fact ("Report upon the atmospheric oxidation of phosphorus, etc.," *Journal Chemical Society*, December, 1880) that the points most dwelled upon by him in this last paper are, that the escaping gas contains no peroxide of hydrogen whatsoever, but consists entirely of ozone, and that "the peroxide of hydrogen formed in the process is entirely deposited in the water contained in the oxidisers; and if it cannot escape condensation in the aqueous spray and vapor of the oxidisers, it cannot be expected to escape solution in five wash-bottles."

Since the statements of Mr. Kingzett now, are diametrically opposed to those which he made when he attacked my results in the beginning, I think it will facilitate the labors of future workers in this field, if I present a brief summary of the facts hitherto established.

When I first entered upon the study of the phenomena exhibited in the aerial oxidation of moist phosphorus (October, 1878), the first difficulty encountered arose from the unsatisfactory character of the



apparatus previously employed, and the impossibility of obtaining a stream of oxidised air containing a uniform and definite percentage of ozone. These difficulties disappeared when it was ascertained that the volume of ozone produced, under given conditions, was a definite function of the temperature, and might accordingly be expressed by a curve, having its origin at 6° C., at which temperature the production was nul, and arriving at its maximum at 24°, from this point falling off again rapidly. The manipulation was rendered easy by the phosphorus ozonator, described along with the volume-temperature experiments (JOURNAL AMERICAN CHEMICAL SOCIETY, I, 8, and *Chem. News*, 40, 157). These results were referred to by Mr. Kingzett in the *Chemical News* for August, 1879, and yet in his earlier experiments performed long subsequently (February, 1880), he states that he obtained no satisfactory evidence of the production of ozone, and but little evidence of the production of peroxide of hydrogen. Later on, when working in summer weather, Mr. Kingzett obtained better results, and discovered that his previous failures had been due to his having conducted the experiments at too low temperatures.

In plotting the volume-temperature curve, the water in the jars of the ozonator was replaced by a solution of potassium bichromate and sulphuric acid of such strength, that the surface of the phosphorus was kept clean without undergoing the danger of ignition from too concentrated acid and too rapid oxidation. Mr. Kingzett states that "such a method of experiment obviously precluded examination for peroxide of hydrogen in this solution, in which the phosphorus was partially submerged," and explained why he found the relation of peroxide of hydrogen to ozone at 1 : 400.

The comment of Mr. Kingzett would have been only too just if I had looked, or proposed to look, in an acidified potassium bichromate bath for undecomposed hydrogen peroxide. But Mr. Kingzett, who criticised both papers at length (*loc. cit.*), must have known at that time, a year and a half ago, though apparently he has since forgotten, that I did not employ such a method. The potassium bichromate bath was employed in the determination of the volume-temperature curve of the evolved ozone only, and in that investigation no attempt was made to determine the hydrogen peroxide formed. That question was taken up at great length in the subsequent paper upon "Ammonium nitrite, and the by-products obtained in the ozonation of air by moist phosphorus." In the experiments therein detailed, and in which the determination of the hydrogen peroxide in the ozonising chambers and in the various wash-waters is express-

ly considered, not merely water, but water especially purified for the purpose, was employed. This and many other precautions were essential at that time, when the exact nature and extent of the sources of error were unknown. And since these sources of error are not even alluded to, nor suitable precautions to obviate them taken by Mr. Kingzett in his paper, it will be necessary to emphasize them here.

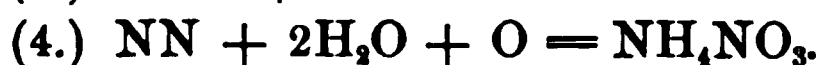
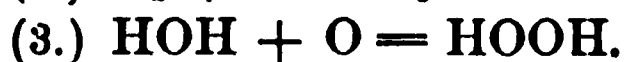
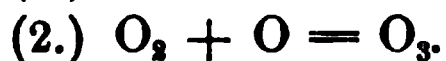
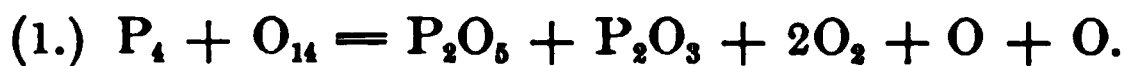
At that time, and even now, the generation of ammonium nitrite by evaporation of water was, and is taught, as an established fact. If this were indeed the case, the ozonising chambers present the most favorable conditions for its generation, and the nitrite thus formed would give the same reaction as hydrogen peroxide or ozone, when we came to titrate the water in the ozonisers with an acidified solution of potassium iodide. The elaborate experiments of Bohlig, Zabelin and Carius, however, have shown that this is not true, and that the alleged formation of ammonium nitrite by evaporation of water in air, never takes place when adequate precautions are taken to exclude from the evaporated water, the ammoniacal compounds pre-existent in the atmosphere. Fortunately, therefore, this source of error was not inherent in the experiments themselves, and it only remained to guard against it by very complete washing of the air drawn through the ozonator.

Again, Goppelsröder and Carius had shown that when ammonia is brought into contact with ozone, it is decomposed with the formation of ammonium nitrite and nitrate, and peroxide of hydrogen. It was essential, therefore, that the phosphorus should be in contact with water from which every trace of ammonia had been expelled. If this were not done, the origin of any ammonium compounds or hydrogen peroxide, found among the products of the reaction, would be rendered doubtful. It was for this reason that all the water employed in my experiments, in which a determination of the hydrogen peroxide was attempted, was *aqua purissima*, that is, water redistilled until it did not give the slightest reaction for ammonia when treated with Nessler's reagent.

Moreover, while the oxidation of water to hydrogen peroxide by *free* ozone had been abundantly disproved, yet there appeared to be much reason for supposing that during the aerial oxidation of moist phosphorus, a formation of hydrogen peroxide actually took place. If this were true, its origin could not be ascribed to a secondary action due to the ozone first formed, but to the same series of changes as those which led up to the oxidation of the oxygen mole-

cule itself. The only sufficient assignable cause was the formation of nascent oxygen, or oxygen in the atomic condition, such as it must necessarily enter into during some moment of the chemical change, when a substance of uneven quantivalence, like phosphorus, undergoes oxidation. That oxygen in an uncombined or monatomic condition, might peroxidise the water molecule, while oxygen already combined in a triatomic molecule might not, appeared to be a legitimate hypothesis. That nascent oxygen might oxidise the nitrogen molecule at the same time, and generate, in the presence of water, ammonium nitrate, I regarded likewise as a not improbable supposition. For this reason, excessive care was taken to preclude the presence of atmospheric ammoniacal or nitrous compounds, both in the air drawn through the ozonator, and also in the water brought into contact with the phosphorus in the ozonator jars.

The mental picture which was formed at that time of the sequence of phenomena to be studied, was embodied in the following equations, the last three being imagined as contemporaneous:



The experiments performed now two years ago, and the repetition of which, though in an imperfect manner and with the neglect of essential precautions, constitute the substance of Mr. Kingzett's report to the Chemical Society above alluded to, were framed with a view of testing the truth or falsity of the above propositions. What these experiments actually were, may be seen by inspection of the accompanying scheme of them, which, with one exception, was followed to the letter :

I. Use of a measured amount of aqua purissima in the ozonator jars.

II. Complete straining and washing of the air by means of cotton wool, aqua purissima, caustic soda and sulphuric acid, both of the latter previously tested and shown to be free from nitrogen compounds.

III. Height of the phosphorus cakes before and after the experiments.

IV. Amount of ammonia in the jar-water at the close of the experiment.

V. Amount of the nitrites and nitrates, as determined by reduction.

VI. Amount of nitrous acid, as determined by metadiamido-benzole.

VII. Amounts of phosphoric and phosphorous acids, and of *hydrogen peroxide in the jar-water* (ozonising chambers).

VIII. Estimation of ammonia, nitrates and nitrites, nitrous acid alone, phosphoric acid, phosphorous acid and hydrogen peroxide, in the water used to wash the escaping products of ozonation.

IX. Similar estimations, except of the phosphorus compounds, in the solutions used to wash the entering air.

X. Measurement of total volumes of air used, and of ozone after its escape from the water employed in washing.

With regard to the first two heads of this scheme, it is to be noted that the *proof* of the exclusion of all compounds of nitrogen from the substance entering into the reaction, was essential to establishing this vital fact—that any nitrogen compounds found among the *products* were the results of chemical changes induced in the process of ozonation itself. That this preliminary proof was of the highest importance was shown by the results of the investigation; it was found that while no ammonium nitrite was present in the water of the ozonising chambers or in the wash-water, of ammonium nitrate a considerable amount (27.39 mgrms) was present in the first, and a determinable quantity (0.31 mgrm) even in the latter. Until adequate experimental evidence, therefore, is adduced to the contrary, the formation of ammonium nitrate during the ozonation of purified air by moist phosphorus, is to be regarded as a fact which cannot be overlooked, and one which demands an explanation in any theoretic account of the chemical phenomena involved.

Mr. Kingzett, however, does not even contemplate the possibility of the nitrogen, which is relatively the most abundant of the substances present, taking part in the reaction. To say nothing of the fact that his arrangements for washing the air were inadequate, there is no proof given that the water used by him in contact with the phosphorus was pure. If it was ordinary distilled water, it certainly was not pure, but contained the very body (ammonia) which was the worst possible impurity, so far as the question at issue was concerned. Not to speak of the discovery of Goppelsröder and Carius, that ammonia is converted by ozone into hydrogen peroxide and the nitrate and nitrite of ammonia. No experiment is narrated by Mr. Kingzett in which he sought to determine whether nitrous acid was present or not, in any of the liquids examined. In the entire absence

of such tests, his determinations of the amounts of hydrogen peroxide, etc., are correspondingly incomplete. That this is not hypocrisy, is shown by the fact that the dense white fumes which are seen in the ozonising chambers, at one time were attributed by Schönbein to ammonium nitrite.

By referring to the seventh head of the scheme, it will be seen that the determination of hydrogen peroxide in the ozonising chambers was expressly mentioned, and as preliminary to this determination, experiments were made upon the possible influence of phosphorous acid upon the titration for the peroxide, and elaborate pains were taken to prove the absence of nitrous acid in the water examined. My surprise was very great on finding, after the publication of the paper, that while the hydrogen peroxide in the wash-water, and every other point in the scheme had been determined, this particular estimation had been overlooked. For this reason, in every communication written subsequently, I have never committed myself to any statement as to the total amount of hydrogen peroxide produced, knowing that it was the sum of three quantities:

1st. That referred to under the seventh head, which remains behind in the jar-water.

2d. That carried forward in a state of aerial suspension, and partly detained by the wash-water.

3d. That escaping along with the ozone in the evolved gas.

Mr. Kingzett has called my attention to the oversight in carrying out this important part of the general scheme, and has made some estimations of the amounts of hydrogen peroxide in the ozonising chambers. Unfortunately, from his use of an acidified solution of potassium iodide as a reagent for titrating the evolved gas, and from his having employed a fan to blow the air through the apparatus without indicating the method of measuring the air, I am unable to use his results.

In my own experiment, in which 56 liters of purified air were drawn through the ozonator, the jars containing pure water, the amount of ozone in the evolved gas was 44.47 mgrms; that of hydrogen peroxide in the jar-water, 35.69 mgrms. This would give for the ratio of the ozone to the hydrogen peroxide, in this particular experiment, 1 : 0.8, or by molecules ( $O_3 = H_2O_2$ ), as 1 : 0.8.

In my earlier paper (August, 1879), the amount of ozone in the evolved gas bore to the amount of peroxide of hydrogen, the ratio of 400 : 1. In these experiments, it was supposed that a long series of wash-bottles was sufficient to wash out the aurally suspended hydro-

gen peroxide, and hence the number, 400 : 1, was taken as expressing the relation between the total quantities in the gas, after its escape from the ozonising chambers. No statement was made, either then or subsequently, as to the total amount of hydrogen produced (owing to my original oversight in failing to determine it in the water of the ozonising chambers), but only to the amount in the evolved gas.

Subsequently, after much more elaborate experiments (*Chem. News*, 42, 19), this assumption was found to be false, and that after passing not only through the wash-bottles, but a length of sulphuric acid dryers, so great that every trace of moisture was removed, the ratio of the ozone to the hydric peroxide in the *evolved gas*, was far less than 400 : 1. In the last and best agreeing series of trials, it was as 51 : 31, or 1 : 0.67. The ratio of the ozone to the water formed by the decomposition of the hydrogen peroxide with the aid of heat (at 200°) was 51 : 18, or approximately 3 : 1. This ratio was accidentally given instead of the former, in stating the result of the trials (*loc. cit.*)

But it will be asked, what proof is there that the neutral solution employed in titrating the current of ozone and hydrogen peroxide, would indicate the ozone only and not the peroxide as well? The answer is to be found in the fact that hydrogen peroxide held in vesicular suspension will pass through neutral potassium iodide solution without causing any appreciable decomposition in the latter. This was verified in the following experiment.\* “Perfectly pure and dry oxygen was ozonised by one of the ‘ozonising elements’ (electrical ozonisers), and then drawn through three bulbs, the first containing a solution of neutral potassium iodide, the second water, and the third chromic acid. The latter was connected with an aspirator. As soon as the strongly ozonised oxygen came into contact with the potassium iodide, a very slow current being employed, a yellow coloration made its appearance on the surface of the solution in the first bulb, and above the surface the characteristic white cloud. On increasing the rapidity of the current, this white cloud was drawn through the water and chromic acid into the aspirator, and remained for one or more hours before its absorption was complete. After drawing over six liters of oxygen containing 0.194 grm of ozone, the water was titrated, and found to have absorbed 0.31 mgrm of peroxide of hydrogen. The chromic acid solution was unaffected, showing that the suspended hydrogen peroxide may pass

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\* JOURNAL AMERICAN CHEMICAL SOCIETY, 2, 157.

through it without effecting a decomposition. The same remark applies to a neutral potassium iodide solution put in the place of the chromic acid. The excessively dilute hydrogen peroxide held in a state of aerial suspension, was not able to decompose the neutral iodide during the course of the experiment. Of course, an *acidified* solution could not be employed to absorb the peroxide, since, as above shown, its decomposition occurs under the action of oxygen alone."

The conclusion, therefore, to be drawn from the above result is, that in the ozonation of air by moist phosphorus, the number of molecules of hydrogen peroxide approximates very nearly to an equality with the number which is, *pari passu*, formed of molecules of ozone. That it does not fully equal the number, may possibly be connected with the production, as the third essential result of the action of nascent oxygen under the circumstances studied, of a certain amount of ammonium nitrate. And not only does this relation exist between the amounts of ozone and hydrogen peroxide *produced*, but subsists with little alteration, after the two bodies have been passed through many wash-bottles and have been, for a considerable interval, in contact with one another in the *evolved gas*.

Now that I have reviewed my own labors in this direction, I wish briefly to examine Mr. Kingzett's statements concerning them.

After incorrectly stating that I looked for hydrogen peroxide in an acidified potassium bichromate bath, Mr. Kingzett adds: "Moreover, it is not apparent from his various communications that he subjected his gaseous product to any very special examination. He seems to have relied upon its odor and general characters rather than upon any exclusive properties, as evidence of its nature."

In the first place, with regard to the odor, Mr. Kingzett has apparently overlooked the fact that, unlike his present position, which is that the body evolved is all ozone, and contains no hydrogen peroxide, he maintained at that time the opposite—that the gas evolved was altogether hydrogen peroxide, and contained no ozone. And the only connection in which I have brought forward the smell of ozone, as an important physical quality, was to state that attention to it might have prevented Mr. Kingzett from so positively affirming that a gas which possessed the powerful and unique smell of ozone, was not ozone, but another body, viz: hydrogen peroxide, which is entirely odorless.

In the next place, Mr. Kingzett has brought forward only two tests, which differ from those which I have applied. The first con-



sisted in passing the ozonised air through turpentine, and assuming that the failure of the gas, after its passage, to set free iodine in a potassium iodide solution, demonstrated that it originally contained ozone only. But until it has been shown that hydrogen peroxide suspended in the gaseous current, would not undergo absorption by oil of turpentine as well, the proof is valueless.

That this absorption would probably take place may be inferred from the action of organic liquids in general, for when the current of ozonised gas is passed into alcohol, ether or glycerine (*JOURNAL AMERICAN CHEMICAL SOCIETY*, I, 448), the white cloud entirely disappears, and hydrogen peroxide is found in the liquid. In this respect the organic liquids strikingly differ from solutions of potassium iodide, water, acidified chromates, etc., inasmuch as the latter do not arrest the suspended hydrogen peroxide.

Mr. Kingzett's second novel test consisted in heating the ozonised air to  $240^{\circ}$ , and assuming that the entire destruction of the active agent at this temperature proved that it was ozone. The information to be derived from heating the gas under proper conditions, is perhaps the most conclusive of any that can be obtained, as to its true nature. But, as Mr. Kingzett has applied the test, he has deprived it of the very conditions essential to its value. For at  $240^{\circ}$ , not only ozone, but also peroxide of hydrogen is destroyed, and there is nothing in the test as he has applied it, to prove that the active agent was ozone only, or peroxide of hydrogen only, or a mixture of both.

Prior to this, I had made use of the same test (*JOURNAL AMERICAN CHEMICAL SOCIETY*, 2, 147, and *Chem. News*, 42, 19), but in a different manner. The ozonised air, after extended washing, was completely desiccated, and then exposed to temperatures running from the ordinary up to  $200^{\circ}$ . It was shown that as the reaction for ozone diminished with the successive increments of temperature, the quantities of water derived from the decomposition of the suspended hydrogen peroxide, as regularly increased. The numbers obtained by experiment exhibited this diminution of ozone and increment of water, as uniformly as was to be reasonably anticipated in view of the difficult nature of the work. The total amount of water obtained in twelve experiments was 0.0086 grm, corresponding to 0.016 grm hydrogen peroxide; the total amount of water obtained in blank experiments continued for a much longer time than those reported, and differing from the latter only in the circumstance that the phosphorus cakes were kept submerged beneath the surface of the water in the ozona-



tor jars, was zero. We have seen with what neglect of essential precautions Mr. Kingzett has applied this test. Against my results he has to urge merely his conviction (?) that they were wrong, and that the amounts of water weighed were due to water only, and not to  $H_2O_2$ . It is hardly worth while to comment—0.016 grm is a ponderable quantity, the reality of which is capable of experimental proof or disproof, whilst a conviction is not necessarily of any weight except in the mind of its author.

The only experiment which Mr. Kingzett has brought forward in this connection, shows that he has failed to apprehend the real question at issue. He passed a rapid current of air through a solution of hydric peroxide, and inferred that the failure of the air, after its passage, to effect a noteworthy decomposition in an acidified potassium iodide solution, proved (what it did not) that no  $H_2O_2$  could be carried forward when the  $H_2O_2$  was in a *state of aerial suspension*. Mr. Kingzett's experiment is a repetition of a similar one of my own published previously (JOURNAL AMERICAN CHEMICAL SOCIETY, 2, 154), but illustrates merely the well-known fact that hydrogen peroxide is a difficultly vaporisable body. Mr. Kingzett appears to think that the use of a *rapid* stream of air had some bearing upon the question, and speaks of a spray of peroxide of hydrogen. The phenomena which are under investigation cannot be studied in this manner. The state of vesicular suspension of the hydrogen peroxide is not one which, so far as we are at present aware, can be brought about by mechanical means, but is one which hydrogen peroxide assumes only when it is the immediate result of a previous chemical change. This is its condition when formed in the phosphorus ozonator, and likewise when ozone undergoes decomposition in presence of water, as when it is absorbed by a solution of potassium iodide, and is the origin of the white cloud seen under these circumstances over the surface of the latter. The difference between the two conceptions Mr. Kingzett would have noted, had he been acquainted with what is the most striking reaction which occurs during the ozonation of air by moist phosphorus. This is the permanent white cloud which fills the ozonator, and is carried forward through an extensive series of wash-bottles, and remains sometimes for hours without undergoing absorption, above the surface of the water in an aspirator employed to draw air through the apparatus. This white cloud was the antozone of Schönbein, which he, and after him, Meissner, labored unsuccessfully for so many years to account for. For our knowledge of its true nature we are indebted mainly to Von Babo, and after him to Nasse

and Engler, who demonstrated that the so-called antozone is merely hydrogen peroxide, in a state of vesicular suspension. The stumbling-block which rendered of so little value the life-long labors of Meissner upon this question was, that starting out from the well-established fact that hydrogen peroxide is not volatile, he concluded that the clouds could not be due to this substance. As the only alternative, he and Schönbein ascribed them to a peculiar modification of oxygen, other than ozone. There is danger of our giving up ground acquired with great difficulty, and falling back into hopeless contradictions, if we do not keep clearly in mind the distinction between hydrogen peroxide in its ordinary condition, in which it is not volatile, and hydrogen peroxide as suspended at the moment of its formation, in a current of air or aqueous vapor. An atmosphere saturated with aqueous vapor is most favorable to the existence of the hydrogen peroxide in a state of vesicular suspension, but is not essential, since after complete withdrawal of the water vapor, the suspended hydrogen peroxide will still be present.

#### SUMMARY.

1st. Both ozone and hydrogen peroxide are produced during the ozonation of purified air by moist phosphorus, in the ratio approximately of one molecule of the latter to one of the former.

2d. Both bodies are evolved, the suspended hydrogen peroxide passing through a series of wash-bottles, without undergoing any but a slight absorption, and being present in the evolved gas in nearly the same ratio as that which it held to the ozone, when originally produced.

3d. Along with these two bodies, and as a necessary part of the same series of reactions, incident originally to the setting free of nascent oxygen, a certain amount of nitrate of ammonia is invariably produced. This last is altogether detained in the water of the ozonator and of the wash-bottles.

#### IV.—UPON THE ACTION OF OXYGEN, OZONE AND NASCENT OXYGEN UPON BENZENE.

BY ALBERT R. LEEDS.

##### I.—OXYGEN.

The first set of experiments was instituted to determine whether oxygen alone, in sun-light or diffused light, or oxygen together with platinum black, in sun-light or diffused light, would oxidise benzene after long-continued exposure. In every case a negative result was obtained, except in that of oxygen and platinum black brought into contact with benzene and a little water in a liter flask, and exposed to the direct sun-light. In the course of a few hours the liquid became slightly yellow, and at the end of eight days was dark yellow and intensely acid. A large amount of oxalic acid had been formed, but no acetic acid and no phenole.

##### II.—OZONE.

The earliest experiments upon the action of ozone upon benzene were made two years ago with the object of further studying the remarkable products obtained by Houzeau and Renard in the course of their investigation upon this subject.\* Along with small amounts of formic and acetic acids, they obtained a gelatinous body which, when dried in vacuo, formed a white amorphous solid, and which they called ozobenzene. It was permanent only at low temperatures, and on gentle warming exploded with tremendous violence. Fearing the possible formation of this dangerous substance, the first experiment was made upon 1 gram of benzene, but the amount operated upon was gradually increased, until in the eighth and final trial, it amounted to 35 grms. In some experiments, the benzene was cooled by a freezing mixture; in other trials, it was ozonised at common temperatures, and in the last, the temperature was kept at 50°. The benzene was exposed to the oxygen, strongly ozonised by the silent discharge and containing about 70 mgrms of ozone in the liter, either in shallow dishes placed under a receiver through which the perfectly dry ozonised gas was kept flowing, or in Geissler bulbs connected with other bulbs to arrest the volatile products of ozonation. But in every case the results were the same. Large volumes of carbonic acid were given off, and the benzene, as it diminished in vol-

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\* *Compt. Rend.*, 76, 572.

ume, became converted into a dark yellow, oily mass, containing crystals of oxalic acid. In other cases, when the ozonation was performed in bulbs, an amorphous blackish mass separated out on the sides of the vessels. From these residues no crystalline bodies could be obtained either by the use of solvents or by sublimation. No indications whatsoever were noted of the white explosive body spoken of by Houzeau and Renard.

Besides carbonic and oxalic, formic and acetic acids were obtained, the latter in considerable quantity. Especial care was paid to the detection of phenole, but as far as our tests were concerned, no evidence of the presence even of a trace of this body was obtained. Hydrogen peroxide was likewise absent.

### III.—NASCENT OXYGEN.

The fact that triatomic oxygen ( $O_3$ , ozone or active oxygen) is not capable of oxidising water to the form of peroxide of hydrogen, while monatomic oxygen ( $O$ , or nascent oxygen) is, rendered it probable that a similar difference would be observed in the effects of ozone and of nascent oxygen, upon benzene. This supposition has been confirmed by experiment. The nascent oxygen was obtained by the action of phosphorus, partially submerged beneath the surface of water, upon air at common temperatures. When no benzene is present, the nascent oxygen oxidises the oxygen molecule to the form of ozone, the water molecule to hydrogen peroxide, and the nitrogen molecule in the presence of water, to ammonium nitrate. But on the addition of benzene, the ozone reaction disappears. The resulting products differ, according as the reaction takes place in sun-light or in diffused light. In the former case, after prolonged exposure, large amounts both of phenole and oxalic acid are formed, the phosphorus is converted into yellowish-red amorphous phosphorus, and the liquid remains colorless. In the latter case, the flask being kept in a warm place, the liquid becomes of a dark-brown color, and abundance of oxalic acid, but no phenole, is formed. At common temperatures the phosphorus goes into solution in the benzene, and is gradually converted into a mass of white dodecahedral crystals, the liquid remaining colorless. It is only when the temperature is above the melting point of the phosphorus (in these trials,  $50-70^\circ$ ) that the dark brown liquid is produced. Curiously enough, though the flask becomes filled with dense white fumes, no inflammation took place at these elevated temperatures, the benzene vapor apparently protecting the phosphorus from ignition. Excess of calcium chloride was added to

the sirupy liquid, the dense precipitate of calcium phosphate and oxalate (somewhat dark from a portion of the organic matter carried down at the same time) filtered off, and the filtrate evaporated to dryness. The soluble portions were then dissolved out with the least possible amount of water, the excess of calcium precipitated out by potassium sulphate, the whole evaporated to dryness, and treated with absolute alcohol. As a small portion of the potassium chloride was taken up by the alcohol, this extract was again evaporated to dryness, and the operation repeated three times, when only the organic matter remained in solution. It was of a yellow color and pasty consistence, but as it could not be made to crystallize, and furthermore was in small amount, its further investigation was abandoned.

After my own research was in progress, I found that Prof. F. Hoppe-Seyler,\* starting out from similar views as to the effects resulting from the action of nascent hydrogen upon oxygen, had performed a series of interesting experiments with hydrogenised palladium foil. A piece of such foil shaken up along with benzene, water and air, in a flask, and allowed to stand for a time, yielded an amount of phenole sufficient, after its conversion by the addition of bromine water into tribromphenole, for analysis. Besides the phenole, Hoppe-Seyler obtained a body which turned brown on the addition of potash.

On repeating the experiment, I obtained a very abundant reaction for phenole. On addition of lime-water, the coloring matter which had been formed at the same time, was thrown down as a yellowish-brown precipitate. Not a trace of oxalic acid was present.

To my own mind, no satisfactory explanation could be given of the formation of phenole under these circumstances, except upon the supposition of the production of peroxide of hydrogen as an intermediate step. In the case of phosphorus oxidising in moist air, the contemporaneous formation of both ozone and peroxide of hydrogen, by the action of the nascent oxygen upon the oxygen and water, is a demonstrated fact.

Three suppositions therefore were admissable. Either the phenole was formed by the direct action of the nascent oxygen ; or 2d. By the action of the ozone, or 3d. By the action of peroxide of hydrogen. The first supposition requires that the oxygen should produce by direct synthesis a molecule of phenole, which is in the highest degree improbable. The second supposition is inadmissable, since

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\* *Ber. der deutsch. Chem. Gesell.*, 12, 1551.

the non-formation of the phenole by treatment with ozone had been abundantly proven. The third supposition admitted of the rational explanation expressed by the formula,



Two modes of testing the validity of the third hypothesis presented themselves. The first was that of bringing the hydrogenised palladium foil into contact with water and air in a flask, and ascertaining if the oxygen rendered nascent by oxidation of the hydrogen, would oxidise the water to the form of hydrogen peroxide. In case an affirmative result was obtained in this experiment, it would be important to try the second test of treating benzene directly with a peroxide of hydrogen solution.

A strip of palladium, after remaining for several hours as the negative pole of a battery of six Bunsen elements, in water acidulated with sulphuric acid, was placed along with 10 c.c. of ammonia-free water in a half-liter flask filled with air. At the end of three days the water was tested with guaiacum and solution of diastase. It developed a deep blue color. On addition of cadmium-iodide-starch solution, iodide of starch was formed, and on titration with sodium hyposulphite, it was found that the total quantity of hydrogen peroxide present in the 10 c.c. of water amounted to 0.67 mgrm.

It is not improbable that both ozone and ammonium nitrate are likewise formed in the course of this reaction, but of the former no satisfactory indication was obtained, and the latter was not looked for, inasmuch as it would have required the repetition of the experiment on a much larger scale and with many additional precautions.

The successful issue of this experiment encouraged us to try what otherwise would not have been attempted, the treatment of the benzene with  $\text{H}_2\text{O}_2$ . 20 c.c. of benzene were allowed to stand at common temperatures for two days in contact with 300 c.c. of a slightly acidulated 1.2 per cent. solution of hydrogen peroxide. The liquids remaining colorless, the flask was attached to a reflux condenser and placed in a water-bath. After the water in the bath had been kept boiling for several days, it was saturated with chloride of calcium, and the digestion continued at this temperature for two days longer. At the end of this time the liquid had become yellow, and was found to contain beside the coloring matter, oxalic acid and phenole. A very abundant precipitate was thrown down from the aqueous solution on addition of bromine water, and this precipitate of tribromphenole, after recrystallisation from alcohol, was found to weigh 0.25 grm.

In conclusion, not only is the direct conversion of benzene into phenole by the action of hydrogen peroxide, of much interest, phenole having hitherto been obtained only by some indirect process, but it supplies a satisfactory demonstration of the validity of the third hypothesis above stated.

## V.—NEW AZO COLORS BELONGING TO THE SO-CALLED TETRAZO GROUP.

BY JAMES H. STEBBINS, JR.

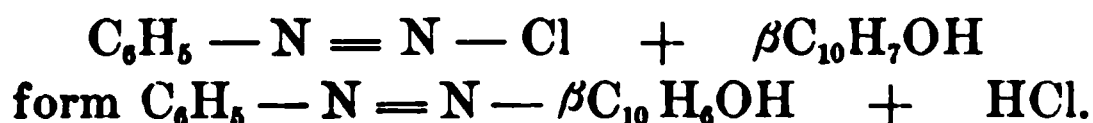
In order to give a clear understanding of this wonderful class of dyes, we will have to go back a few steps and consider the ordinary diazo compounds, their formation, and the azo dyes derived from them.

It is a pretty generally known fact, that when any of the salts of aniline (as the chloride, nitrate, etc.) or its homologues are treated with nitrous acid (either free or in the shape of a nitrite), that diazo-benzole chlorides, etc., are formed, viz :



Now from Griess we learn that diazo compounds unite directly with phenoles and amines to form coloring matters which belong to the class of the so-called azo compounds.

As an example of the combination of diazo compounds with phenoles, we may take the action of diazobenzole chloride upon beta naphthole, viz :



If, on the other hand, we allow diazobenzole to react upon an amine (as aniline), we would get an amido-azo compound,



and this is the basis of the new class of colors.

Amido-azobenzole is a yellow basic coloring matter, uniting with one molecule of acids to form salts which, however, are readily decomposed with boiling water.

Now, as we have seen how nitrous acid acted upon amines, it seemed only too probable that if amido-azobenzole chloride were treated with nitrous acid, that a diazo compound would be formed, inasmuch as it contains a free amido radical ( $\text{NH}_2$ ). This hypothesis was fully verified by Kékulé, who describes such a compound in his organic chemistry. This discovery, at that time, presented no value,



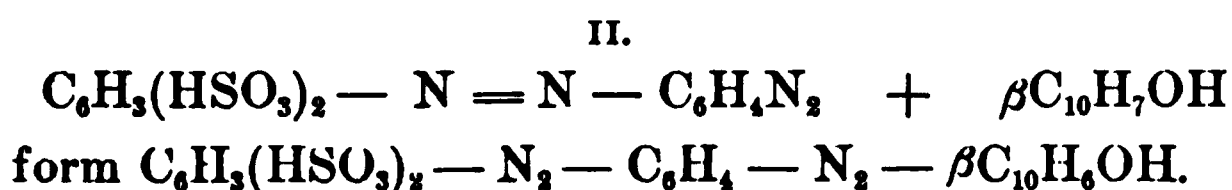
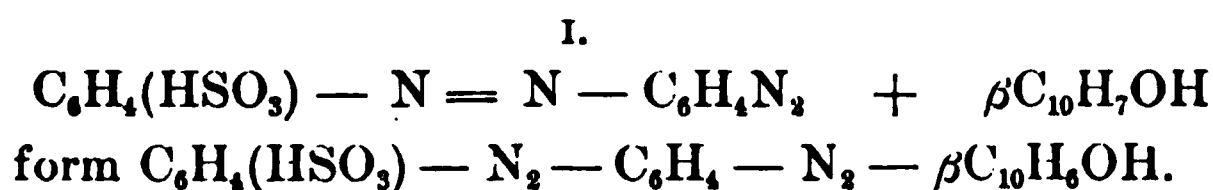
as azo compounds derived therefrom were insoluble in water, and could not be used for technical purposes.

Things remained in this state till a few years since, when it was discovered, that by introducing the sulpho group ( $\text{HSO}_3$ ) into these compounds, they were rendered soluble in water and might be used for dyeing.

Based upon this theory, Graepler took out a patent for converting the hitherto useless amido-azobenzole into its sulpho acid, and since then the manufacture of the tetrazo dyes has made great progress.

One of the first parties to avail themselves of this invention was the firm of Calle & Co., in Biebrich, who shortly after sent into commerce a dye-stuff called "Biebrich scarlet," and this dye is produced by diazotising amido-azobenzo-sulpho acids, and combining the so obtained diazobenzo-sulpho acids with beta-naphthole, in an alkaline solution.

I say sulpho acids, because, in the preparation of amido-azobenzo-sulpho acid, not only the mono, but also the disulpho acid are formed, and we therefore have a mixture of two dye-stuffs as the final result, viz:

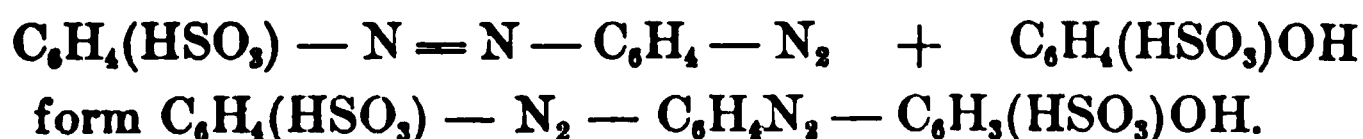


We therefore see, that wherever an aromatic azo compound contains a free amido group, that such a compound is capable of being diazotised, and again combined with phenoles to form new coloring matters.

Basing my experiments on this theory, I have been able to produce a series of new colors, of which the following are examples :

I.

When diazo-azobenzo-sulpho acid is allowed to react on an alkaline solution of phenol-sulpho acid, a strong yellow dye-stuff is the result.

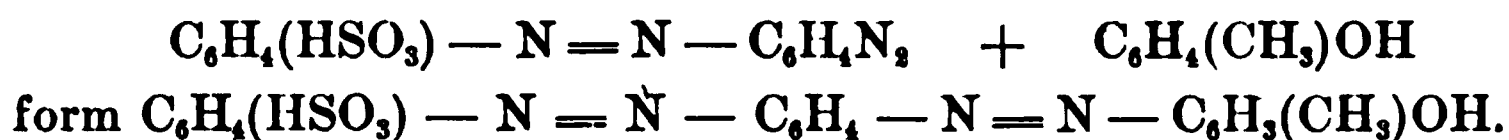


This color is very soluble in water, and dyes wool of an intense yellow color in an acid bath.



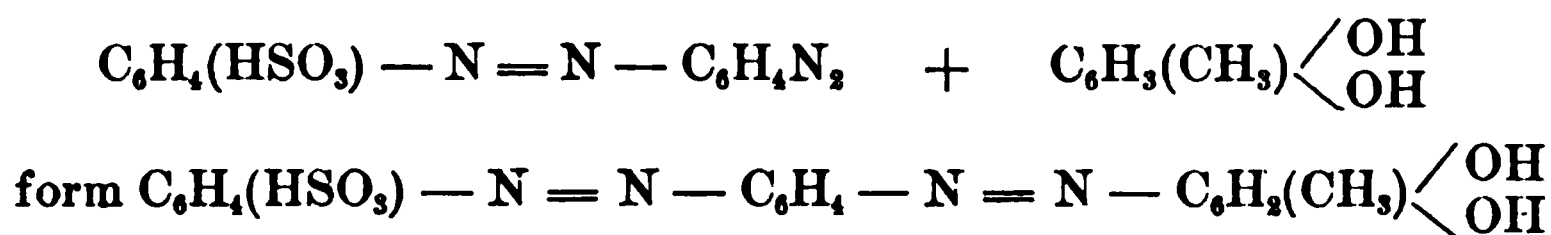
## II.

If diazo-azobenzo-sulpho acid is allowed to react on cresole or its sulpho acid, a yellow coloring matter is likewise formed, which does not materially differ from the previous compound.



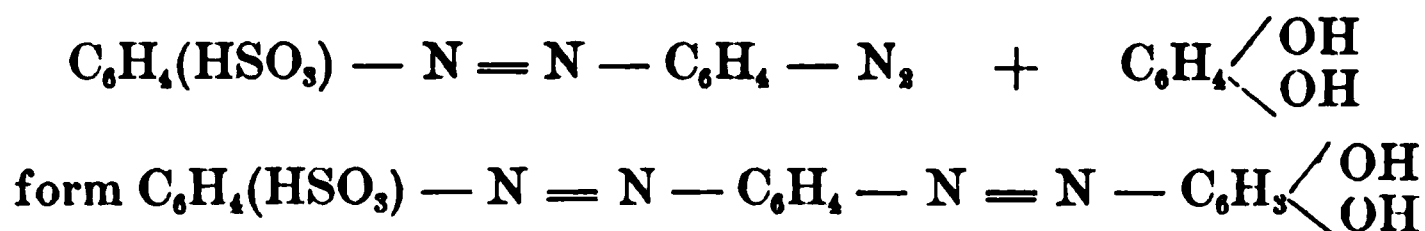
## III.

An alkaline solution of orcinol produced an orange color.



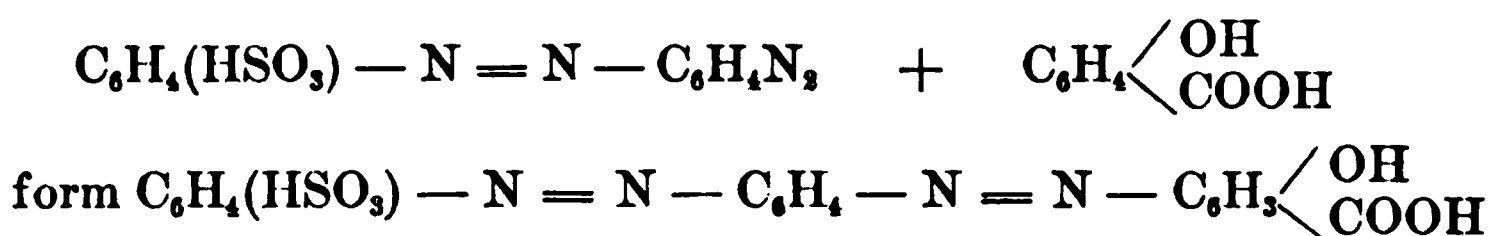
## IV.

Resorcinol treated in the same way produced likewise an orange dye-stuff.



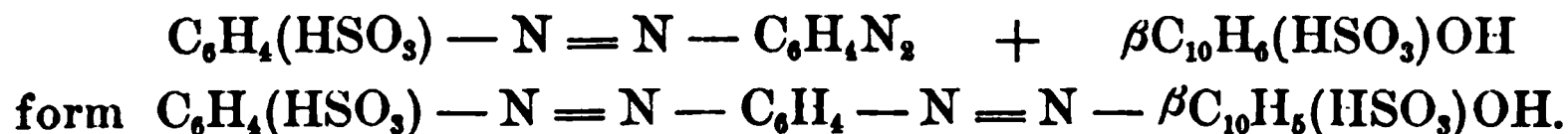
## V.

Salicylic acid brought in contact with diazo-azobenzo-sulpho acid produced a yellow dye-stuff.



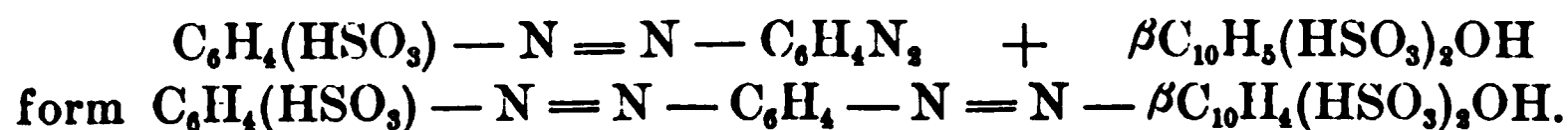
## VI.

In the same way a fine scarlet color is obtained, if beta-naphthol-sulpho acid is substituted for salicylic acid.



## VII.

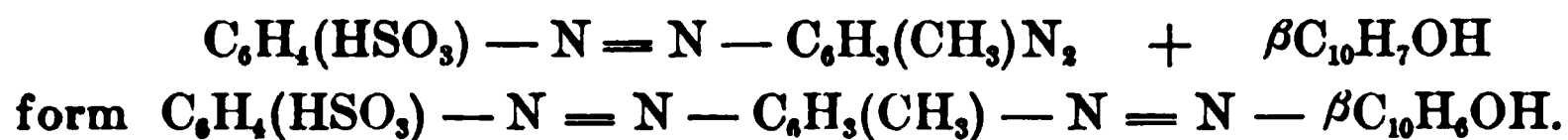
If we substitute beta-naphthol-disulpho acid for the monosulpho acid, a fine red color is the result. (Pat. by Koehler.)



## VIII.

Finally, if we treat azosulphoxybenzodiazotoluene with an alkaline solution of beta-naphthol, an intense red coloring matter is

obtained. This coloring matter is very similar to Biebrich scarlet, but has a somewhat bluer shade.



These dyes are nearly twice as strong as the ordinary azo dyes, and they may be distinguished from one another by a very sharp reaction. When such a dye-stuff contains the sulpho group ( $\text{HSO}_3$ ) in the benzole nucleus, it is converted into an intense green color by the addition of a few drops of strong  $\text{H}_2\text{SO}_4$ . When the sulpho groups are in both the benzole and naphthole nuclei, an intense blue color is obtained, but, when only in the naphthole nucleus, a purple color is the result.

We can thus tell almost at a glance, the nature of the color under examination.

# THE AMERICAN CHEMICAL SOCIETY.

## VI.—PROCEEDINGS.

### *Regular Meeting, March 7th, 1881.*

At 8:30 P.M. the Society was called to order by Vice-President Dr. E. R. SQUIBB, who presided.

Sixteen persons were present at the time.

The minutes of the January meeting were read by the Recording Secretary. Adopted.

The minutes of the February meeting were read by the Recording Secretary. Adopted.

The following gentlemen resigned their membership :

Mr. ELIHU ROOT,	Mr. H. J. SCHMITZ,
Dr. T. J. O'CONNOR,	Mr. FLORIAN ALEXANDER.

Their resignations were adopted unanimously.

Mr. CASAMAJOR announced that Dr. GALLATIN had resigned his office as Recording Secretary. The resignation was accepted with regret.

It was moved that a committee of one be appointed by the chair to select a new Recording Secretary. Carried unanimously. Mr. CASAMAJOR was appointed as said committee.

The following gentlemen were nominated as new members :

J. G. MATTISON.

Nominated by { P. DE P. RICKETTS,  
ARTHUR H. ELLIOTT,  
C. F. CHANDLER.

THEO. TONNELÉ.

Nominated by { P. DE P. RICKETTS,  
ARTHUR H. ELLIOTT,  
C. F. CHANDLER.

Accepted unanimously and referred to Committee on Nominations.

Dr. BEHR then read the first paper announced for the evening, as follows :

“A New Specific Gravity Bottle.” By WM. H. GREGG.

Dr. SQUIBB, in discussing the paper, described the specific gravity bottle used by him. Mr. CASAMAJOR described the specific gravity bottle which he employed.

Dr. BEHR then read the second paper announced for the evening, as follows :

“On the Solvent Action of Carbonic Anhydride in Solution, upon Various Bodies, under Different Conditions as to Temperature and Pressure.” By T. H. TUCKER.

Dr. SQUIBB remarked that water at a sufficiently high temperature decomposes bicarbonates in solution. There is a dissociation in a narrow limit of temperature, and the carbonate of the salt is deposited.

Mr. CASAMAJOR announced that he had received a paper from Dr. THOMASSIE on the “Dissocioscope.” He stated that this paper had already been published in the *Chemical News*. Referred to the Committee on Papers and Publications.

Mr. CASAMAJOR read a paper on the production of sulphuretted hydrogen by electrolysis, caused by the presence of zinc amalgam and sulphide of iron.

Mr. STEBBINS then read a paper “On Certain New Diamido-compounds.”

Dr. LEEDS then made some remarks on the action of peroxide of hydrogen on ammonia. The ammonia is oxidised, producing nitrous acid.

Then followed a discussion by several members on different methods of testing for nitric and nitrous acids, in presence of each other.

Dr. LEEDS moved that hereafter the nights on which the regular meetings and the conversaziones were held, should be changed to Friday. The regular business meeting to be held the first Friday of the month, and the conversazione on the third Friday of the month. Carried unanimously.

Mr. CASAMAJOR moved that the Society have an annual dinner, the time thereof to be fixed by a committee to be appointed. Carried unanimously.

Mr. CASAMAJOR moved that the Chair appoint a committee for the next annual dinner. Carried unanimously.

The Chair appointed as said committee Mr. CASAMAJOR and Dr. ALSBERG. Adopted unanimously.

On motion the Society adjourned.

ALBERT H. GALLATIN,  
*Recording Secretary.*

## VII.—A NEW SPECIFIC GRAVITY BOTTLE.

By DR. W. H. GREGG.

In taking the specific gravity of corrosive and volatile fluids, the ordinary specific gravity bottle presents some objections. The stoppers are usually perforated, with a view of affording a full passage for the excess of fluid when the stopper is inserted into its place. This is very objectionable with corrosive fluids, such as oil vitriol, and such volatile fluids as ether, chloroform, etc.



For such substances, a modification of Regnault's specific gravity bottle is presented, which does not allow any overflow of its contents when the stopper is inserted, and at the same time a thermometer is at hand to note the temperature. Such a bottle is shown in the figure. The neck is drawn out as in Regnault's, and marked to indicate its capacity. A short distance above the mark the neck swells out sufficiently to contain an amount of fluid equal to that displaced by the thermometer tube. The thermometer is attached to the stopper which is ground into the neck at the top as on the ordinary bottle, to prevent loss by evaporation.

Elmira, N. Y.

## VIII.—ON THE SOLVENT ACTION OF CARBONIC ANHYDRIDE IN SOLUTION, UPON VARIOUS BODIES, UNDER DIFFERENT CONDITIONS AS TO TEMPERATURE AND PRESSURE.

By J. H. TUCKER, PH.D.

It is well known that carbonic acid gas in solution, is capable of exerting a great chemical influence upon the materials forming the earth's crust, and as the air and all natural waters are impregnated with it, the range of its action is necessarily extensive. Among the bodies thus acted upon by carbonic acid, may be mentioned the carbonates and silicates as the most important. The insoluble carbonates are dissolved, soluble bicarbonates being formed, the extra equivalent of carbonic acid being given up again on heating the solution. The silicates are decomposed, the base is converted into carbonate or acid carbonate, the silica remaining insoluble; the decomposition or "rotting" of feldspathic rocks is a prominent example of this. In addition to the compounds mentioned, the tribasic phos-

phates of lime and magnesia, the fluoride of calcium and phosphate of alumina, are known to be soluble in a solution of carbonic anhydride. It has been claimed that silica in the hydrated state is soluble, but the results of my experiments with ferrous silicate, as far as they go, seem to negative that claim. There is good ground, however, for believing that a large number of the constituents of rock masses are decomposed by carbonic acid, and it will be the chief object of an extension of this investigation to examine these decompositions, and determine their quantitative relations.

As far as I am aware,\* the only work that has been done on this subject in a quantitative way, has been principally by Bischoff (*Chemical and Physical Geology, English Ed.*), who has limited his experiments mostly to the carbonates of lime and magnesia, which were performed only at a single temperature and pressure. Lassaigne (*Journ. Chim. Med.*, 1848, 312) states that water saturated with carbonic acid, dissolves  $\frac{1}{145}$  part of calcic carbonate at  $0^{\circ}$  C., and  $\frac{1}{115}$  part at  $10^{\circ}$  C.; and Warrington (*Journal Chemical Society*, 2, 9, 80) states that one part of calcium phosphate in calcined bone-ash, dissolves on the average in 6788 parts of the saturated water.

In view of the importance of the *role* played by carbonic acid in geological changes, and the modifying influence which we know differences of temperature and pressure exert upon its action, I have thought it would be useful to make quantitative determinations of the solvent action on various bodies at varying temperatures and pressures. The following is the method adopted to carrying on the experiments: At ordinary pressure, the purified gas was passed into distilled water containing a large excess of the substance under examination, in a minute state of division, the passage of the gas being continued for six or eight hours until it ceased to be absorbed.

For the pressure effects, the finely divided substance was diffused through water; the gas passed at ordinary pressure for an hour, and then the whole exposed to a pressure of one atmosphere for six to eight hours, the containing vessel being frequently agitated to aid the absorption of the gas. The results obtained at  $0^{\circ}$  C. were by artificial cooling with ice.

#### FERROUS CARBONATE.

I. *At  $0^{\circ}$  C.*—1. Well washed hydrated ferrous carbonate, only slightly oxidised, was diffused through distilled water and the gas passed:

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\* Schloesing, *Journ. Chem. Soc.* 2, 10, 788; Cossa, *Zeitschr. f. Anal. Chem.*, 1869, 145.

After  $1\frac{1}{2}$  hours, one liter of solution contained 1.308 gm  $\text{FeCO}_3$ .

"	3	"	"	"	"	"	1.632	"	"
"	$4\frac{1}{2}$	"	"	"	"	"	2.332	"	"
"	6	"	"	"	"	"	2.594	"	"

2. In another experiment, the gas was passed for  $4\frac{1}{2}$  hours when one liter of solution contained 2.512 grms  $\text{FeCO}_3$ .

II. *At ordinary temperature.*—Conditions as above :

Time, eight hours. One liter of solution contained :

At $23^\circ \text{C.}$ ,	1.046 gm $\text{FeCO}_3$ .
At $21^\circ \text{C.}$ ,	1.134 " "

III. *Under pressure.*—One liter of solution contained :

(1.)	1.520 gm $\text{FeCO}_3$ .
(2.)	1.374 " "

*Siderite.*—A well crystallized specimen was reduced to fine powder and treated as above at  $24^\circ \text{C.}$ , under atmospheric pressure. One liter of solution contained : 0.480 gm  $\text{FeCO}_3$ .

#### CALCIUM CARBONATE.

I. *At  $0^\circ \text{C.}$* —The carbonate used was formed by precipitating a solution of calcium chloride.

One liter of solution contained : 2.444 grms  $\text{CaCO}_3$ .

II. *At ordinary temperature.*—One liter of solution contained at  $28^\circ \text{C.}$ , 0.894 gm  $\text{CaCO}_3$ .

III. *Under pressure.*—One liter of solution contained at  $21^\circ \text{C.}$ , 1.1824 gm  $\text{CaCO}_3$ .

IV. *Marble.*—Ordinary crystalline marble was finely powdered and submitted to the action of the gas :

At  $22^\circ \text{C.}$ , and under ordinary pressure, one liter of solution contained 1.036 gm  $\text{CaCO}_3$ .

#### MAGNESIUM CARBONATE.

I. *At  $0^\circ \text{C.}$*

II. *At  $30^\circ \text{C.}$* —One liter of solution contained 10.676 grms  $\text{MgCO}_3$ .

III. *Under pressure.*—One liter contained, at  $26^\circ \text{C.}$ , 18.520 grms  $\text{MgCO}_3$ .

The carbonate operated upon was obtained by precipitating a solution of magnesian sulphate at boiling temperature, with sodium carbonate.

## ANKERITE.

(Carbonate of lime, magnesia and ferrous oxide.)

Native ankerite consisting of the powdered crystals, was treated with carbonic acid gas at 25° C., in the usual way, under ordinary pressure.

One liter of solution contained 0.318 grm  $\text{CaCO}_3$ , 0.313 grm  $\text{MgCO}_3$ , with a little  $\text{FeCO}_3$ .

It will be observed that the relative amounts dissolved of the calcium and magnesium salts, are very different from those dissolved when the salts are operated upon separately.

## MANGANESE CARBONATE.

Artificial hydrated Carbonate.

*At 0° C.*—One liter of solution contained 0.396 grm  $\text{MnCO}_3$ .

*At 26° C.*—One liter of solution contained 0.239 grm  $\text{MnCO}_3$ .

*Under pressure, at 23° C.*—One liter of solution contained 0.376 grm  $\text{MnCO}_3$ .

## TRIBASIC PHOSPHATE OF LIME.

In the hydrated state, prepared by precipitating a solution of calcium chloride with sodium orthophosphate.

*At 25° C.*—One liter of solution contained 0.396 grm anhydrous phosphate.

*Under pressure, at 28° C.*—One liter of solution contained 0.464 grm anhydrous phosphate.

## PURE HYDRATED ALUMINA

Exposed to the action of the gas in the usual manner, showed not the slightest solubility under any temperature and pressure.

## HYDRATED SILICATE OF PROTOXIDE OF IRON

Prepared by precipitation, was exposed to the action of the gas at 23°C. and ordinary pressure.

One liter of resulting solution contained 0.330 grm  $\text{FeCO}_3$ .

On careful testing of the same solution, no trace of silica was found.

From these results it may be observed :

1. That difference in temperature produces a marked difference in the amount carried into solution ; the lower the temperature, the more being dissolved.

2. That increase in pressure causes a considerable increase in matter dissolved, though the effect is not so great as in the case of



change in temperature ; carbonate of lime seems to be an exception to this, increased pressure appearing to make little difference.

3. Substances in the native state, as compared with those artificially prepared, are, as would be expected, less soluble, but they are sufficiently soluble to fully account for the profound geological changes that have been produced by carbonic acid gas.

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### IX.—NOTE ON SULPHURETTED HYDROGEN.

BY P. CASAMAJOR.

In 1873, Mr. William Skey, Analyst to the Geological Survey of New Zealand, published some interesting observations\* on the production of sulphuretted hydrogen, when a voltaic couple is formed with zinc as the positive, and a metallic sulphuret as the negative plate. When placed in contact with zinc, or in communication with this metal, by means of a wire, many sulphurets give sulphuretted hydrogen very freely, with sulphuric or hydrochloric acid, which alone would not give any in contact with these acids. This happens with galena and sulphuret of copper.

I have had occasion lately to apply these researches of Mr. Skey to the production of sulphuretted hydrogen from sulphuret of iron, which refused to give it in the presence of sulphuric acid diluted with ten times its volume of water. I believe that the greater part of the sulphuret of iron sold to chemists possesses this resistance to acids. I have found none lately which gave sulphuretted hydrogen easily, and I had accumulated several pounds lately possessing this undesirable quality.

After a few trials I was able to obtain an abundant supply of sulphuretted hydrogen from any sample of sulphuret of iron, by proceeding as follows: Enough mercury is put in a bottle to cover the bottom entirely. Over this diluted sulphuric acid is poured, and some pieces of zinc are thrown in, which immediately sink in the mercury, forming a zinc amalgam with great excess of mercury. No action takes place between the zinc and the acid. If now a few pieces of sulphuret of iron are thrown in the bottle, they will sink to the mercury, and a copious discharge of sulphuretted hydrogen will take place from the surface of the sulphuret of iron. This production will continue with remarkable regularity until either the zinc, the sulphuret of iron, or the sulphuric acid is exhausted. As to the mercury, it

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\* *Chemical News*, 27, 161.

only intervenes indirectly in the reaction, and it does not require to be renewed.

In place of sulphuret of iron I have tried galena, iron pyrites and copper pyrites. These all give sulphuretted hydrogen, but very slowly. With galena the action soon ceases as the mineral becomes coated with sulphate of lead. By leaving galena in contact with the zinc amalgam for several hours, its surface becomes coated with bright lead amalgam.

With iron and copper pyrites comparatively little sulphuret of hydrogen is given off, but there is a considerable production of hydrogen. In this case the sulphurets act mainly as conductors, in the same manner as a piece of retort carbon.

# THE AMERICAN CHEMICAL SOCIETY.

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## X.—PROCEEDINGS.

### *Regular Meeting, April 1st, 1881.*

At 8:20 P.M. the meeting was called to order by Vice-President LEEDS.

Twelve persons were present.

Dr. LEEDS stated that all business would be deferred until a quorum should be present.

In absence of Mr. PIRKIN, the second paper of the evening: "On Anilometallic Compounds" was read by Dr. LEEDS.\*

Mr. STEBBINS stated that he had made an aniline and calcium compound in the presence of water.

The Recording Secretary announced that a quorum was now present.

The minutes of the March meeting were read by the Recording Secretary. Adopted after sundry corrections had been made.

The following gentlemen were nominated as new members:

A. HOWARD VAN LINDEREN, associate member.

Nominated by { ARTHUR H. ELLIOTT,  
T. O'CONNOR SLOANE,  
J. B. HERRESHOFF.

C. P. SAWYER, associate member.

Nominated by { ARTHUR H. ELLIOTT,  
ELWYN WALLER,  
P. CASAMAJOR.

Dr. OTTO GROTE.

Nominated by { BERNARD G. AMEND,  
S. A. GOLDSCHMIDT,  
JAS. H. STEBBINS, Jr.

Mr. A. E. HOPPOCK, of 103 East 148th street, was unanimously elected a member of the Society.

Mr. CASAMAJOR, who had been appointed a committee of one at the last meeting to select a new Recording Secretary, announced that he had selected Mr. JAS. H. STEBBINS, Jr., as Recording Secretary. It was moved and recorded that this report be adopted. Carried unanimously.

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\*Withdrawn by the Author.

Mr. CASAMAJOR then announced that he had selected Mr. J. B. HERRESHOFF to take the place of Mr. STEBBINS on the Committee of Nominations. It was moved and seconded that this report be adopted. Carried unanimously.

Mr. CASAMAJOR then made the report of the Committee on the Annual Dinner.

It was moved and seconded that the annual dinner should take place on Saturday evening, the 16th day of April, at 5 p.m. Carried.

Mr. PITKIN read his paper entitled: "Action of Concentrated Sulphuric Acid on Lead Alloys."\*

A long discussion followed participated in by various members.

Mr. ARTHUR H. ELLIOTT moved to reconsider the action on the motion to have the annual dinner on Saturday evening, the 16th of April. Seconded and carried.

It was then moved and seconded that we do hold our annual dinner on Monday evening, the 18th of April, at 6 p.m. Carried unanimously.

On motion, the Society adjourned.

ALBERT H. GALLATIN,  
*Recording Secretary.*

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\* This paper has, at the time of publication, not been received by the Com. on P. and P.

# THE AMERICAN CHEMICAL SOCIETY.

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## XI.—PROCEEDINGS.

*Regular Meeting, May 6th, 1881.*

The meeting was called to order at 8:30 P.M., Prof. LEEDS in the chair.

In the absence of a quorum, the regular business was deferred till one should be present.

The first paper of the evening, "On a Slight Modification of Wilkinson's Gas Eudiometer" was read by Mr. JAS. H. STEBBINS, Jr., S.B.

Prof. DOREMUS then remarked that the trouble encountered with the Wilkinson eudiometer might be overcome by first filling with gas and then letting in a little water, and finally making allowance for the water level.

The second paper of the evening, "On the Purification of Baric Sulphate," by T. O'CONNOR SLOANE, Ph.D., was then read.

The third paper of the evening, "On a Qualitative Test for Carbon Disulphide and Carbon Dioxide in Coal Gas," by T. O'CONNOR SLOANE, concluded the reading of papers.

The Recording Secretary then announced that a quorum was present.

The minutes of the last meeting were then read and approved.

Mr. CASAMAJOR objected to the mention of particulars concerning annual dinner, and thought that only a general description in place of minute particulars would be preferable.

Dr. SQUIBB then stated that he thought that the minutes concerning the annual dinner as taken by the Recording Secretary, should not be altered, but, that minute particulars should be left out of printed report.

This being put to motion, was carried unanimously.

Dr. GOLDMARK's death was then mentioned by the chair.

Dr. SQUIBB moved that a committee of one be appointed to draw up a short obituary notice on Dr. GOLDMARK's death.

On motion, this was carried unanimously.

The Recording Secretary then read the minutes of the last meeting of the Board of Directors, which, after a small correction, were approved.

The Recording Secretary then read Dr. MOORE's resignation as Editor of the JOURNAL OF THE AMERICAN CHEMICAL SOCIETY.

After sundry remarks by Messrs. SQUIBB, LEDOUX and CASAMAJOR, Dr. DOREMUS moved that a committee, consisting of Drs. A. R. LEEDS and A. R. LEDOUX, be appointed to report in regard to filling vacancy caused by Dr. MOORE's resignation.

This being put to motion, was carried unanimously.

The chair then brought up the question in regard to filling the vacancy in the Board of Directors, caused by the death of Dr. GOLDMARK.

After some remarks by Messrs. SQUIBB, ELLIOTT and CASAMAJOR, it was finally moved and seconded that the previous committee, consisting of Drs. A. R. LEED and A. R. LEDOUX, should also investigate the legality of the election of a Director to fill the vacancy in the Board of Directors, caused by the death of Dr. GOLDMARK, and should report at the next meeting, presenting at the same time the name of a person to fill the vacancy, and that the Recording Secretary should insert a notification to this effect on next postal cards.

On motion, this was carried unanimously.

The following gentlemen were then elected :

C. P. SAWYER, associate member.

Nominated by { ARTHUR H. ELLIOTT,  
ELWYN WALLER,  
P. CASAMAJOR.

A. HOWARD VAN LINDEREN, associate member.

Nominated by { ARTHUR H. ELLIOTT,  
T. O'C. SLOANE,  
J. B. F. HERRESHOFF.

Dr. OTTO GROTE, as member.

Nominated by { S. A. GOLDSCHMIDT,  
BERNARD G. AMEND,  
JAMES H. STEBBINS, Jr.

THOS. TONNELÉ, as member.

Nominated by { P. DE P. RICKETTS,  
ARTHUR H. ELLIOTT,  
C. F. CHANDLER.

J. G. MATTISON, as member.

Nominated by { P. DE P. RICKETTS,  
ARTHUR H. ELLIOTT,  
C. F. CHANDLER.

Mr. A. P. HALLOCK, Ph.B., was then nominated as a regular member, by T. O'C. SLOANE, Mr. BENJAMIN and Wm. RUPP.

The fourth paper of the evening, "Upon the Direct Conversion of Aromatic Amides into their Corresponding Azo-compounds," by Dr. A. R. LEEDS, was then read.

After some questions by Mr. STEBBINS, the fifth paper of the evening, "On the Chemical Contents of the Fluid Cavities of Minerals," by Mr. A. A. JULIEN, was then read.

Some remarks by Messrs. ELLIOTT and DOREMUS then followed. On motion, the Society adjourned.

JAMES H. STEBBINS, JR.,  
*Recording Secretary.*

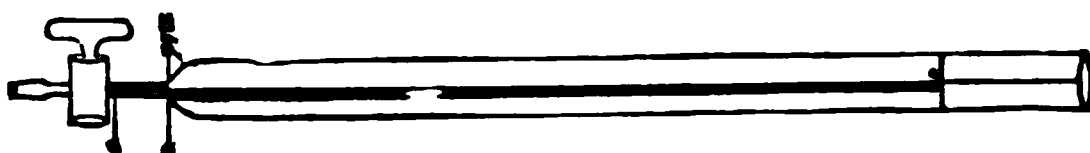
## XII.—ON A SLIGHT MODIFICATION OF THE WILKINSON GAS EUDIOMETER.

By JAMES H. STEBBINS, Jr.

At one of our previous meetings, Prof. Doremus read a paper and exhibited to you one of Wilkinson's eudiometers, with which, I think, he said very fair gas analyses might be made.

Now, although I do not wish to contradict Prof. Doremus' statement, still I feel bound to say, that my experience with the above apparatus for the past month has been anything but satisfactory, and I now make bold enough to say that the Wilkinson gas eudiometer, as it is now made, cannot possibly give correct results, and with the greatest care, only approximate answers can be obtained.

The reason of this is obvious and can readily be explained by the following diagram :



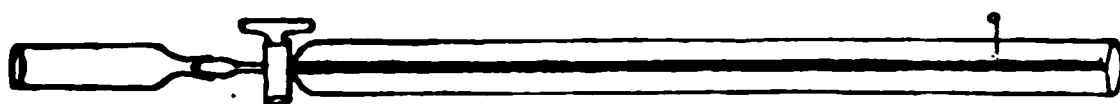
The usual Wilkinson eudiometer has quite a long and thin neck just under the glass cock as may be seen in the diagram, and which I represent as *a* and *b*.

Now, in filling this burette, we have to first fill it with water, which drives all the air out, and then connect it with the rubber tube conveying the gas. As soon as we open the cock, the gas flows in, which we so regulate as to get down to the zero mark, and then turn off the valve. In this way we get just 100 c.c. of gas. Now

the trouble begins, for the moment we let our absorbing fluids flow in, the narrow neck of the eudiometer is immediately filled with the liquid, and naturally drives the gas down below the zero mark, thus causing much trouble and annoyance. Now Prof. Doremus will say that this difficulty can easily be overcome by first letting pure water run into your burette, then marking the expansion caused, and finally making allowance for this expansion after we begin to absorb our different constituents.

But I will here remark that, as soon as our water level has fallen below zero, it is almost impossible to make accurate readings with the naked eye, which can only be done with a telescope having cross hairs, and such a thing as this for quick work is entirely out of the question.

Therefore, to get rid of all this trouble, I have done away with the long neck, and have had a eudiometer made with the glass cock soldered directly on to the top of the burette.



This burette is divided into 100 c.c. and  $\frac{1}{10}$  of c.c., thus permitting me to make very close readings.

The *modus operandi* of this instrument is the same as in the previous case, and the analyses obtained are approximately correct.

The modification here mentioned may be better understood by the following diagram :



### XIII.—1. NOTE ON THE PRECIPITATION AND PURIFICATION OF BARIUM SULPHATE. 2. QUALITATIVE TEST FOR CARBON DISULPHIDE AND CARBON DIOXIDE IN COAL GAS.

By T. O'CONNOR SLOANE, A.M., PH.D.

#### 1.

To obtain a precipitate of barium sulphate that will not run through the filter paper, the following rules should be observed : The solution should be barely acid. This end is best secured by the use of cochineal or some equivalent solution. By using the colored matter in the fluid form, a solution can be neutralized in quarter of the time, and with far more exactness than when its reaction is tried



with litmus paper. The precipitant is added when the solution is nearly boiling, and the liquid is kept at that temperature for some time. A heavy precipitate will be formed with perfectly clear supernatant liquor. It is quite certain, if any iron be present, that some will be carried down, and the ignited barium sulphate will not be white. Many ways of treating the precipitate after ignition have been recommended. I will refer to only two in the hurried way. The first is, to boil it with dilute hydrochloric acid ; the second, to boil it with concentrated sulphuric acid ; then dilute and filter. If the first course be pursued, any barium sulphide present will be dissolved, thus causing a loss. This is quite a common case, as the filter paper reduces the sulphate in the ignition. If treatment with concentrated sulphuric acid be adopted, any other salts of barium present will be rendered insoluble, and excess of weight be caused. In the dry way, fusion with sodium carbonate and a little sodium nitrate, and subsequent determination of the sulphuric acid, may be recommended as very efficacious ; but it is troublesome.

Some time ago I had occasion to make a great many sulphur determinations in coal. I was much annoyed by the precipitation of iron with the barium sulphate. I adopted the following simple method of purification, and it always secured a perfectly white precipitate :

The sulphuric acid was precipitated with great care, as already described, so as to obtain a clear supernatant fluid. The solution was decanted to the last drop through a filter paper. To the precipitate left in the beaker were added five or ten cubic centimeters of concentrated hydrochloric acid. The beaker was then carefully held over and just touching a hot plate, until the acid was in full ebullition. By holding the beaker in an inclined position there will be no danger of spirting. The boiling was kept up for about a minute. After cooling, it was diluted, cochineal solution added, overneutralized with ammonia, slightly acidified and filtered. This always gave perfectly white precipitates.

## 2.

A piece of caustic potash a few mms long is dissolved in 10 to 20 c.c. of alcohol. If, after standing protected from the air, a homogeneous and clear solution remains, it is ready for use. Otherwise, after settling, the clear fluid may be decanted. It is placed in a suitable absorption tube and a cubic foot or more of gas passed through it. It is then poured into a test tube. If the gas contained carbonic acid, an oily-looking colorless solution of potassic carbonate

in water will underlie the alcohol. This last will be colored more or less.

The alcoholic solution, if any carbon disulphide was in the gas, will contain a so-called xanthate of potash, and may be boiled and tested for sulphuretted hydrogen.

A more demonstrative method is to add excess of copper salt, filter out the precipitated cupric compounds, and pour ammonia through the filter paper, when a highly characteristic precipitate of cupric xanthate will remain on the filter paper.

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#### XIV.—UPON THE DIRECT CONVERSION OF THE AROMATIC AMIDES INTO THEIR CORRESPONDING AZO-COMPOUNDS.

BY ALBERT R. LEEDS.

I have already published in another place (JOURNAL AMERICAN CHEMICAL SOCIETY, 3, 16,) the results obtained by the action of hydrogen peroxide upon benzene. The latter is gradually converted into phenole-oxalic acid, and a small amount of a yellow coloring matter being formed at the same time.

Phenole itself is but slightly acted upon by hydrogen peroxide in the cold, but at the boiling point undergoes conversion into a tarry substance.

Naphthalene, when digested with hydrogen peroxide, yields a small amount of naphthole. Naphthole itself is not affected to any notable extent by hydrogen peroxide in the cold, but at the boiling point is changed to a tar.

When anthracene is dissolved in glacial acetic acid, and the precipitate obtained on the addition of solution of hydrogen peroxide is digested with excess of the latter re-agent, the anthracene is slowly converted into anthraquinone. Three grms of anthracene, boiled for three days with hydrogen peroxide, in a flask attached to a return-cooler, were changed almost entirely into anthraquinone, the brown coloring matter formed at the same time being too small in amount to permit of its being experimented upon successfully.

Benzenesulphonic acid was slightly acted upon, a brown color being imparted to its solution after boiling for a number of hours with hydrogen peroxide, and the acid crystallising out from the liquid in small colorless crystals, identical in appearance with the original body before its treatment with peroxide.

The preceding results show that in certain cases hydrogen peroxide operates upon organic bodies by the substitution of hydroxyl; in other cases, by direct oxidation, and in the third place, by both actions combined.

The experiments were therefore extended to the aromatic amides with the hope that hydroxylated derivatives of these compounds might be obtained. The amide was converted into the acetate, or when this not possible, it was dissolved in excess of glacial acetic acid, and a solution of hydrogen peroxide, containing 1.2 per cent. of peroxide, added in successive portions. Heating of the liquid had to be avoided, the amide in every instance being changed to a tarry substance. This result indeed happened in most instances, even when the experiment was conducted at common temperatures or with gentle warming. The following is a brief summary of the results obtained with the compounds thus far operated upon :

Diphenylamine yielded a large amount of a black liquid and a black tar.

Dimethylaniline gave a very dark red liquid with a black residue soluble in alcohol.

Xylidine formed a claret colored liquid with a blackish residue dissolving in alcohol to a dark purple solution.

Naphthylamine gave a rose colored liquid with a brownish-black residue.

Similar results were obtained with orthotoluidine, the solution acquiring a ruby color, and a blackish tarry residue soluble in alcohol being formed.

Failing to obtain by careful sublimation, or by treatment with ligroine, alcohol and other solvents, any crystalline bodies from the above products, their further study was abandoned.

Aniline and paratoluidine acted differently. The solutions of their acetates in hydrogen peroxide quickly threw down on gentle warming, copious precipitates of a brownish crystalline appearance. At higher temperatures there was a formation of tar. The precipitates came down principally on the first treatment with the peroxide. On filtering them off and adding an amount of the peroxide in excess of the calculated quantity, the second crop of crystals was small, and the larger portion of the aniline or paratoluidine acetate remained behind in the solution.

The precipitates, after washing and drying, were sublimed very slowly, since the small amounts of accompanying tarry matter interfered seriously with the operation when the temperature was not carefully regulated at the lowest practicable point. The sublimate from the paratoluidine formed yellow needles, some of them exceeding two inches in length. They were repeatedly crystallised from alcohol, in which they are not very readily soluble, and were finally

obtained as magnificent orange needles. Their melting point was 143–144°.

0.2806 grm of the substance yielded on combustion 0.8123 grm carbonic acid and 0.1763 grm water. These results agree with those calculated on the supposition that the body is azotoluole,  $C_{14}H_{14}N_2$ :

	Calculated.	Found.
Carbon	80.00	79.28
Hydrogen	6.66	6.98
Nitrogen	13.34	13.51

The same body has recently been obtained by Barsilowsky\* by the action of very dilute potassium permanganate solution upon toluidine sulphate. This reaction yielded a very small product, and Barsilowsky afterwards used a solution of potassium ferricyanide and caustic potash with better results. The orange crystals thus obtained melted at 144°, and were identical in their physical properties both with the parazotoluene, derived by Petrieff from the reduction of nitrotoluene, and with the substance described by the author. By the action of hydrogen peroxide, therefore, upon paratoluidine it is converted directly into parazotoluene.

The sublimate from the aniline product yielded large red crystals, together with some accompanying yellow oil. This sublimate had a melting point of 66°, showing that the body was azotoluene (m. pt., 66°.5).

## XV.—ON THE EXAMINATION OF CARBON DIOXIDE IN THE FLUID CAVITIES OF TOPAZ.

BY ALEXIS A. JULIEN.

### *Literature.*

- SIR DAVID BREWSTER. See for ref., J. D. Dana, system of Mineralogy, articles Brewsterlinite and Cryptolinite; also *Phil. Mag.*, 1847, 3, 31, 497; 1853, 4, 5, 235, etc.  
 R. TH. SIMMLER. *Pogg. Ann.*, 1858, 105, 460.  
 H. VOGELSANG and H. GEISSLER. *Pogg. Ann.*, 1869, 137, 56.  
 ISAAC LEA. *Proc. Ac. Nat. Sci.*, Phil., May, 1876.

Recent studies of the fluid contents of the cavities of minerals, especially of the fluids separated in sufficient quantities for analytic examination, have established more definite views concerning their true character. The liquid first observed by Brewster in Brazilian topaz, and which has since passed by the name Brewsterline or Brewsterlinite, has been shown by Simmler, Vogelsang and Geissler,

\**Ann. der Chem.*, 207, 102.

and W. N. Hartley,\* to be identical in its properties with liquid carbon dioxide. The abundant dissemination of this substance has been variously and certainly confirmed through its general optical and physical properties, especially by the determination of its refractive index, by spectroscopic examination of the gas after expulsion into a Geissler tube, and even by ordinary qualitative or volumetric examination.

The dense and darker liquid commonly associated with this, first noticed by Brewster, and afterwards styled cryptoline or cryptolinite, as well as another liquid, still more thick and viscid, subsequently observed in a large cavity,† are now recognised merely as saline solutions of varying composition.

These two names are now obsolete, but the following substances may be accepted as entering into the content of these cavities in the various siliceous minerals : water, liquid and as vapor, and frequently in the form of supersaturated saline solutions, containing chlorides, sulphates, or carbonates of the alkaline metals, fluoride and sulphate of calcium, and sulphate of barium, some of these salts, especially the chlorides, being often isolated in minute crystals ; carbon dioxide, liquid and gaseous nitrogen, solid bitumen, mineral oil,‡ and perhaps lighter hydrocarbons ; free sulphuric and hydrochloric acids ; hydrogen sulphide ; and minute crystals, microliths, and trichites, representing usually the constituent minerals of the enclosing rock, *e. g.*, hornblende, rutile, etc. Sulphurous oxide and free ammonia have been also detected in the gases expelled from the cavities by ignition,§ but may have originated in the decomposition of solid or liquid bituminous matters, such as those first noticed by Foster in smoky quartz ; their actual presence in the included gases is yet to be confirmed by examination of the latter, when separated without adulteration, by products of distillation, *e. g.*, by mechanical means, as when crushed in a vacuum. Several other soluble substances of limited occurrence have been noted in the study of special minerals, *e. g.*, magnesium and calcium chlorides in the cavities of halite, zinc sulphate with sodium chloride in those of blende,|| etc.

In the siliceous sedimentary and metamorphic rocks, especially in all the gneisses of the Appalachians, in the Eastern United

\* *Journ. Chem. Soc.*, London, 1876.

† Brewster, *Phil. Mag.*, 1853, 4, 5, 235.

‡ Brewster, *Trans. Roy. Soc.*, Ed., 10.

§ A. W. Wright, *Amer. Journ. Sci.*, 1881, 3, 21, 212.

|| A. Schertel, *Berg und Hüttenm. Zeitung*, 1878.

States, the quartz grains generally abound in cavities, holding apparently pure water, but sometimes a saline solution with minute cubical crystals. For instance, I have found certain specimens of the triassic brown sandstone of Portland, Conn., to be remarkably rich in these fluid cavities, not only the quartz but even all the feldspar grains being saturated by them to such a degree, that they abound in every field of view throughout the thin section.

Carbon dioxide so far appears to be of much rarer occurrence in the rocks of Eastern America, having been detected mainly in quartz, and only at the following localities: in the labradorite of Labrador; in hornblende-syenite at Columbia, N. H.;\* in the pegmatyte vein at Branchville, Conn.;† in the granulyte at Charlotte, N. C., and in the smoky quartz at White Plains, N. C.;‡ in the hornblende-gneiss of the Laurentian at Ticonderoga, N. Y., and in the same rock at three localities in Western N. C.; in the gneissoid-granite of the Penokie Iron Range, Wis.,§ and in granite, mica-schist, hornblende-schist, and chlorite-schist, at three localities in Mich.||

In the metamorphic and eruptive rocks of Western America, however, carbon dioxide, together with a second liquid, which is either water or a saline solution, occupies the cavities of the quartz grains at many localities—in the gneisses, granite and granite-porphyry of Marble Hill, Kinzly District, of Clover Cañon, Humboldt Mts., of Havallah Range, and of Granite Peak, Ute Range, Nev.; in the hornblende-gneiss at Davis Mt., Park Range, and in the quartz-propylite at Golconda, Col.;¶ in the smoky quartz of Pike's Peak, Col.,\*\* and in the quartz from the gold vein at Hunter's Rest, Arizona.†† These few localities, however, must undoubtedly represent a far wider distribution of this interesting substance throughout the rocks of the continent, as their number is being constantly increased with every published report of lithological work.

The qualitative identification of carbon dioxide in the cavities of a mounted thin section of a mineral, may be determined, at least with probability, after some experience, through various optical appearances and physical characteristics which have been often described.

\* G. W. Hawes, *Geol. of N. H.*, 1878, 3, 207.

† A. W. Wright, *loc. cit.*

‡ G. W. Hawes, *Amer. Journ. Sci.*, 1881, 203.

§ R. D. Irving, *Geol. of Wis.*, 1880, 3, 249.

| A. Wichmann, *idem*, 600, 619, 635, 642, 647.

¶ F. Zirkel, *Geol. Expl. of 40th Par.*, 1876, 6, 18, 33, 44, etc.

\*\* B. Silliman, *Science*, 1880, 1, 289.

†† G. W. Hawes, *Amer. Journ. Sci.*, 1881, 203.

It is usually effected with certainty and ease, through the rapid and enormous expansion and ultimate disappearance, either of the liquid or of the gaseous bubble, on the application of a gentle heat for a few seconds, such as that of a cigar, the heated end of a rod, a jet of hot air, or even a jet of the warm breath conveyed through a flexible rubber tube. When the slide and the thin section are thin, even the heat ( $37^{\circ}\text{C.}$ ) of the tip of one's finger applied for a few seconds to the bottom of the slide, without removal from the stage of the microscope, may be sufficient to produce the characteristic phenomena, *e. g.*, the contraction and disappearance of a bubble whose size is relatively small to that of the liquid in which it floats.

For the determination of the temperature of disappearance of the bubble, which may vary from  $20^{\circ}$  to  $32^{\circ}\text{C.}$ , several forms of stage-heating apparatus may be employed; those of Nacet, L. Beale, and R. Fuess, in which a current of hot air from the flame of a small lamp or taper is conveyed beneath both the slide and the adjacent bulb of a thermometer; that of M. Schultze (also Chevalier, Dujardin, and Dr. Ransom), in which both slide and thermometer bulb are heated by conduction through a brass or copper plate, with two arms projecting beyond the stage over the flames of two tapers; those of Polallion and Ranvier, in which the slide rests upon the upper side of a metal box through which a continuous current of hot water is conveyed through tubes in connection with a small adjacent tank or boiler, the bulb of the thermometer being immersed in the box; and that of H. Vogelsang, in which the slide rests upon a ring-shaped thermometer bulb, with a fine and frequently bent platinum wire intervening, which is heated by a galvanic current under the control of a rheostat. The results obtained by any apparatus of these kinds are inexact, from the unequal and irregular application of heat to the thin section and to the thermometer bulb, from the interference of cooler currents in the surrounding atmosphere, and above all, from the refrigerating effect of the mass of metal of the microscopic objective in close approximation. Vogelsang admitted that with his apparatus, the one even yet considered the most accurate, from this last source the error may amount to  $10^{\circ}$ , according to the objective employed, from a No. 4 Hartnack, of 3 mms focal distance, to a No. 9 of 0.1 mm focal distance. He reduced observations with the higher powers to the standard of the No. 4, and further suggested a plus correction of  $1^{\circ}$  for those observations in which the temperature of the room and of the microscope remained at  $12^{\circ}$  to  $15^{\circ}$ , instead of his normal,  $20^{\circ}\text{C.}$  Most of the published observations on the subject



have been consequently made under the lowest powers, and it is considered sufficient to obtain results corresponding to a degree. In every case there are also additional obstacles in the inconvenience, fragility and cost of the apparatus ; much time is required for satisfactory experiments, and especial annoyance is incurred by the slowness with which the apparatus cools before the temperature of the re-appearance of the bubble can be determined, as a check.

In place of all these a simple and inexpensive apparatus may be substituted, consisting of a miniature water bath in which are immersed the entire section and slide, the bulb of the thermometer, and the nose of the objective. It consists of a box of tinned copper

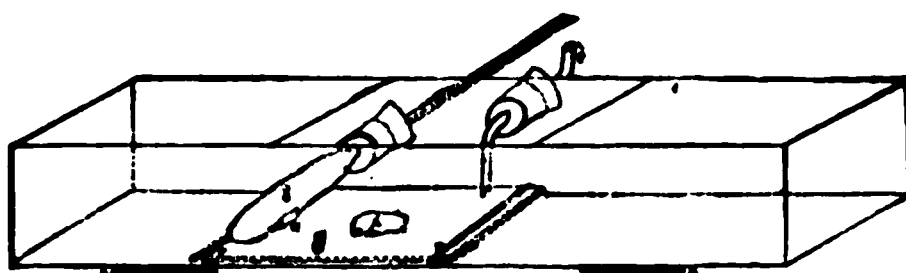


Fig. 1.

(tinned iron is liable to rust), of length sufficient to project a few centimeters on either side of the stage of the microscope employed ; the one I use being 23 centimeters in length, 4 cms in width, and 3 cms in depth. This is laid across the stage of the microscope, separated from the metal by thin plates of cork, *c c*, and is heated by a short wax taper (night light) underneath either extremity. The slide *s* may rest upon the bottom guarded from the metal by little rubber bands, *r r*, beneath its ends, and wedged firmly by a little wooden wedge, *w*, beneath the horizontal thermometer bulb, *b* ; or, a thermometer with ring-shaped bulb may be inserted, upon which the slide may rest directly, firmly attached by one or two slender rubber bands. The thermometer should be of guaranteed accuracy, with wide degrees, subdivided if possible, with a range which need not much exceed 20 to 32° C. The preparation is then covered by any pure and clear water, preferably filtered (distilled is unnecessary), to a depth of about 2 cms. A circular aperture in the bottom of the box 18 mms in diameter, is covered with glass attached by cement, and through this the light is thrown up from the mirror. The cavity to be examined is then carefully adjusted and focussed, a taper is lit, and the eye remains at the eye-piece until the critical point is reached. The glass tube *t*, with its point terminating just below the edge of the slide, is connected with the mouth during the experiment by a small rubber tube. As the temperature slowly



rises, a constant current of small bubbles of the warm breath (whose temperature,  $32^{\circ}$ , only assists the operation) may be blown with little fatigue through the tube to effect a thorough intermixture of unequally heated layers in the water stratum. The determination of the temperature of disappearance of the bubble is easily obtained within five minutes, and that of its re-appearance in about the same time. A low power objective may be carefully wiped if its anterior lens is dimmed by flying drops or by rising vapor, when a high temperature is being attained ; but it is best to insert the whole objective in a small narrow glass beaker floating upon the surface of the bath over the preparation.

The plan of immersing the preparation at once in a bath previously heated, and then allowing it to cool under observation, has already been used with indifferent results. Brewster "plunged the topaz in heated water," and thus determined the point of re-appearance of the bubble at  $26^{\circ}.4$  C., and, in another deep cavity, at  $28^{\circ}.8$  C. Vogelsang placed it in 6 to 8 c.c. of glycerine, previously warmed to  $60^{\circ}$ , in a layer 4 to 5 mms in depth above the preparation ; this he stirred up with a slender thermometer, which, however, greatly interfered with observation. He obtained the following results, corresponding to the Hartnack objectives employed, for the temperature of re-appearance of the bubble :

Objective No. 4, ocular No. 3, lens above the liquid	. 31 $^{\circ}$ .5-(32)
" " 4, " " 4, " just in the liquid	. 34 $^{\circ}$ .5-(36)
" " 7, " " " hardly down to	. 45 $^{\circ}$ .

As the range of error amounts to  $13^{\circ}$ , even without the use of an objective of much power, he recommends the employment in this method of such an objective as will remain above the liquid, but considers his galvanic apparatus as preferable. Sorby\* explains that he examined the fluid cavities in sapphire, by immersing the preparation in a beaker of water, under a low power of the microscope, and raising its temperature very slowly, but gives no details concerning the method or the accuracy of his results. A bath may, indeed, be heated up by repeated additions of small quantities of warmer water, but this process is apt to be tedious and slow. With my apparatus above described, an accuracy within one-tenth of a degree was readily and constantly obtained, both with a one inch and a one-eighth inch immersion objective. It will suffice to present, out of a large number of unusually good results on a fluid cavity of 0.54 mm diameter,

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\* H. C. Sorby and P. J. Butler, *Proc. Roy. Soc.*, London, 1869, 17, 297.

those obtained by means of the Fuess apparatus (*F*), the one most commonly used, and others by means of my own (*J*), the temperature being noted both of the disappearance (*d*) and re-appearance (*r*) of the bubble. The objective used was a one inch of Wm. Wales, with a working distance of 13 mms in the air, and 15 mms over a stratum of water 7 mms deep.

	<i>F.</i>	<i>J.</i>		
<i>d</i>	26.7	26.5	26.5	26.5
<i>r</i>	26.4	26.3	26.4	26.4

In place of the blowing tube a little agitator or churn at the end of the box had been previously tried, to produce the intermixture of the contents of the bath, but was given up on account of the inconvenient vibration and frequent dislodgement of the slide.

The apparatus, as thus constructed, I have long used, and may be found the most convenient warm stage when high temperatures are required; but another still more simple, lately devised, will best serve for the determination of carbon dioxide, and consists of the following parts :

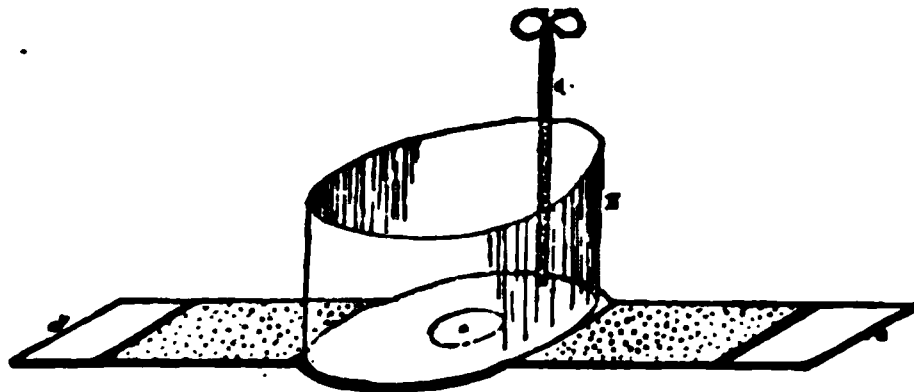


Fig. 2.

*First.* A shallow glass tank with thin and well annealed sides, of size sufficient to enclose the slide upon which the thin section is mounted. For this purpose I use a small chemical beaker *B*, with the thinnest bottom, and with its upper portion cut off, forming a thin round glass tank, about 6 centimeters in diameter and 3 cms deep.

*Secondly.* A plate of copper or brass like that used in Schultze's apparatus, or more simply one of the form represented in the figure, *d e*. Its dimensions, proportioned to those of the beaker-tank and of the stage of a large microscope, are as follows ; length, 23 cms ; diameter at center, 6.5 cms ; width of arms, 3.5 cms ; central aperture, 2.5 cms ; height of wire support, 13 cms ; thickness of plate, 1 mm. Each arm is wrapped in pasteboard, to prevent radiation, to the extent indicated by the shaded portion.

*Thirdly.* A delicate thermometer (made by Henry Green, of New York), with a small short bulb bent at right angles to the stem, and a very fine column, to obtain sufficient sensitiveness to minute varia-

tions of temperature, and complete immersion of the bulb in the small volume of liquid employed in the bath. The scale need not exceed in range from about  $20^{\circ}$  to  $32^{\circ}$  C., the thermometer being of such length that, when in position, the scale from  $27^{\circ}$  to  $30^{\circ}$  C. may be on the level of the eye-piece of the microscope, and readily visible without motion of the head. Each degree of the column should be about a cm in length and subdivided to tenths. The instrument used was standardized, and had a plus error of about  $0^{\circ}.05$  at the portion of the scale employed.

*Lastly.* A pointed glass tube, with flexible rubber connection for blowing, and a wire support, *s*, to receive both this and the thermometer, attached to the metal plate.

The latter is laid upon the stage of the microscope, separated by thin plates of cork or a perforated piece of pasteboard; the tank, supplied with about 40 c.c. of water, is placed over the central aperture, *a*, and a taper beneath an extremity of one arm of the plate, and the apparatus is then ready for use in the way already described; the water of the tank being heated by conduction through the metal plate. The section of the mineral is best mounted upon a very thin slide, 45x26 mms, and this is guarded as before by rubber bands and held down by one or two little brass weights. Only a single taper is necessary for the low temperature required in the examination of carbon dioxide cavities, and even with this, a temperature of  $43^{\circ}$  C. may be obtained in the bath within a few minutes. The disappearance of the bubble may be completed in less than five minutes, the taper being removed as soon as the rising column approaches within 2 or 3 degrees of the critical point, roughly determined by a previous trial. If two tapers are used, the temperature of the water may be raised to  $55^{\circ}$  in about 20 minutes, or even much higher by the use of Bunsen gas burners. In summer the temperature of the atmosphere alone may be sufficient, especially if assisted merely by the current of warm breath, to obliterate the gas bubble. Its return may be readily caused, in a warm atmosphere, by adding from time to time a few drops of cool water to the bath, while the eye remains at the eye-piece, and a steady current of air is blown through the glass tube. Mounted slides used for such experiments must be labelled by writing with a diamond, or the paper label may be rendered waterproof by being coated successively with weak size and any transparent varnish, such as copal or shellac. To ascertain the limits of accuracy, the following determinations were made of the temperatures of disappearance (*d*) and re-appearance (*r*) of the bubbles in two cavities,

*A* and *B*, of the same thin section, with a variety of objectives, on two stands made by Fuess, of Berlin, and Powell & Lealand, of London, the objective being usually immersed in water, but sometimes not immersed. For comparison, the experiments of Vogelsang were exactly repeated with this apparatus, using two objectives of the same kind immersed in glycerine.

Objective.	Cavity A.			Cavity B.		
	<i>d</i>	<i>r</i>	Mean.	<i>d</i>	<i>r</i>	Mean.
Hartnack, No. 2	28.30	28.00	28.15	27.60	27.50	27.55
“ “ 4	28.40	28.20	28.30	27.60	27.70	27.65
“ “ 4, in glyc.	28.32	....	28.32	27.95	....	27.95
“ “ 7	28.40	28.10	28.25	27.85	27.80	27.82
“ “ 7, in glyc.	28.28	....	28.28	27.80	....	27.80
“ “ 9	28.35	28.32	28.33	27.80	27.60	27.70
W. Wales, 3 inch, not imm.	28.40	28.35	28.37	27.97	27.92	27.94
“ “ 1 “	28.40	28.35	28.37	27.96	27.92	27.94
“ “ 1 “ not imm.	28.39	28.38	28.39	27.65	27.80	27.72
“ “ $\frac{1}{2}$ “	28.35	28.37	28.36	27.96	27.96	27.96
“ “ $\frac{1}{16}$ “	28.10	27.90	28.00	27.80	27.82	27.81
Pow. & Leal., $\frac{1}{8}$ “ imm.	28.35	28.36	28.36	27.72	27.70	27.71
Average	....	....	28.33	....	....	27.79

From these experiments it may be inferred that with this apparatus, which may be called the immersion warm bath, it matters little for most purposes what liquid, stand or objective is employed; that water is preferable to glycerine from its greater mobility, convenience, and lack of cost, but its bulk is immaterial, so long as the bulb of the thermometer is covered; that it is decidedly advantageous to immerse the anterior lens of every objective in the bath, to avoid the annoying interference with observation produced by the vibration of the surface, and by the necessity for repeated re-focussing, when the objective is above the surface of the liquid; that careful determinations on minute cavities, with high powers, carried on slowly to enable the preparation, objective and thermometer to assume the same temperature, may be as accurate as any others; and that there is no difficulty in obtaining satisfactorily the two determinations within ten minutes to an approximation of about one-twentieth of a degree. The influence upon the temperature of the disappearance of the bubble exerted by forced dilatation,\* producing adherence to the side of the cavity, appears to be very small, never exceeding a few hundredths of a degree, probably because the walls of these cavities

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\* Berthelot, *Ann. Chim.*, (3,) 30, 232.

are not dry, but always lined by at least an imperceptible film of another liquid, the brine or saline solution.

The descriptions of this method, and of these forms of apparatus, have been given in the more detail, inasmuch as they may be of service in many other branches of thermal microscopy where the exact determination of the temperature applied is desirable, *e. g.*, as suggested by my friend, Mr. Arthur H. Elliott, in the determination of the melting point of rare chemical substances, etc. For this purpose, the apparatus in Fig. 1 might be supplied with another tube, on the opposite side to those represented, through which might be inserted, beneath the objective, a small glass tube, containing the substance to be examined, and thus immersed, by the side of the thermometer bulb, in the water, oil, paraffine, or other liquid which the circumstances may require for the bath.

The liquid inclusions in the white topaz of Rio Belmonte, Brazil, have been already the subject of considerable investigation by Brewster, and others; but the recent study of a large number of cleavage slices from fifty pebbles of this mineral, labelled "Minas Geraes, Brazil," peculiarly rich in fluid cavities, has presented several facts of some interest which have not been recorded by these observers.

Although it can hardly be claimed that the mysterious spontaneous motion which very commonly affects the gas bubbles in the most minute microscopic cavities, usually but a few thousands of a mm in diameter, has yet received a complete and perfectly satisfactory explanation, there can be little doubt that its essential dependence upon slight changes of temperature\* has been sufficiently demonstrated. This view is confirmed by the fact that in a cavity 0.03 mm in length, in one of my slices of the topaz, the bubble, quiescent at ordinary temperatures, passes into active motion when the temperature is slightly raised, so that observation of this spontaneous motion becomes possible—a unique instance so far as I am aware—with a magnifying power of only 65 diameters. Several other like instances have been noted in these slices.

It is well known that the fluid cavities of most minerals lie to a large extent in planes, and that those of rounded symmetrical form predominate. Beautiful crystalline forms—in quartz, inverted crystals—often occur, and Brewster and Vogelsang have pointed out their occasional presence in topazes, exhibiting the contour of "negative rhombic tables and distorted octahedra." In a few choice slices of

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\* G. W. Hawes, *Am. Journ. Sci.*, *loc. cit.*

my specimens of topaz, however, many extremely angular, elongated, branching and even reticulated forms of considerable size and novelty abound. Their outline is at many points decidedly crystalline, with

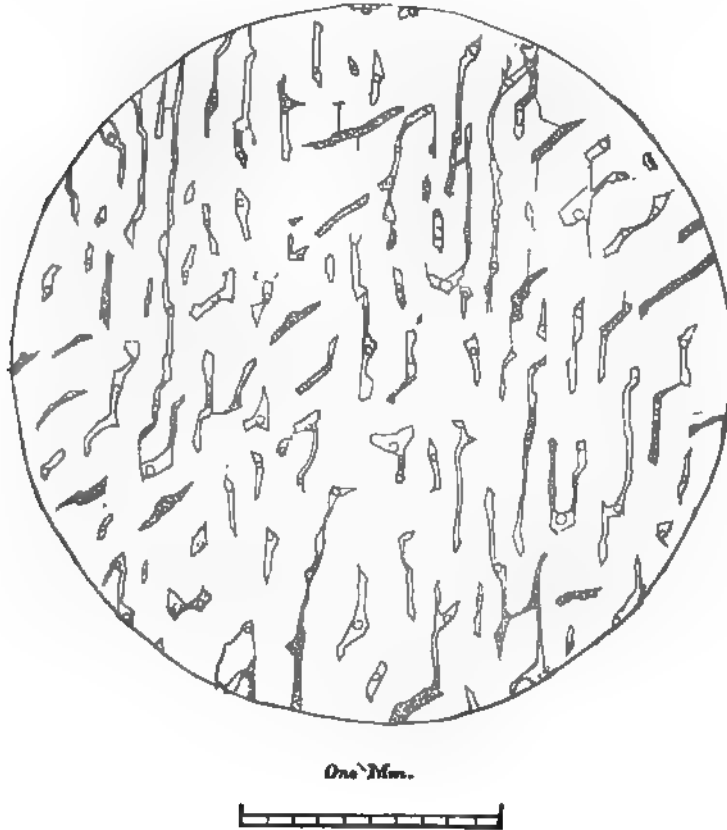


Fig. 3.

arms projecting at an angle of about  $135^\circ$ . As the preparation in which those figures occur, is a natural cleavage slice on the basal plane, this disposition of the cavities seems to have been guided by a parallelism to prismatic faces—it may be, on the planes  $i-i\Lambda i-2 = 136^\circ 35\frac{1}{2}'$ . If so, the generalization of Brewster may have been pressed too far, that the cavities were generally “capriciously distributed, when the substance of the crystal was in a soft or plastic state.” There is also a curious partial division of the two liquids, in regard to distribution in the cavities, the limpid and colorless carbon

dioxide, generally including a bubble, being mainly concentrated in the longer tubes, vertical in the figure, with a little brine at their extremities, while the thick and dark saline solution is mainly confined to the arms which project at the angle stated, and fills completely, always without bubbles, those which are straight, short, and lie entirely in that direction. This may indicate the result of molecular pressure in two directions, differing greatly in degree upon two liquids of widely differing density, the lightest and most mobile having been squeezed into the plane of least pressure. In general, the larger expansions of the cavities are mostly occupied by brine, while their attenuated extremities and fine tubular connections are filled by liquid carbon dioxide, occasionally including a bubble due to contraction.

The intricate connection of adjoining tubes may even produce cavities of unusual length, so that one containing both brine and

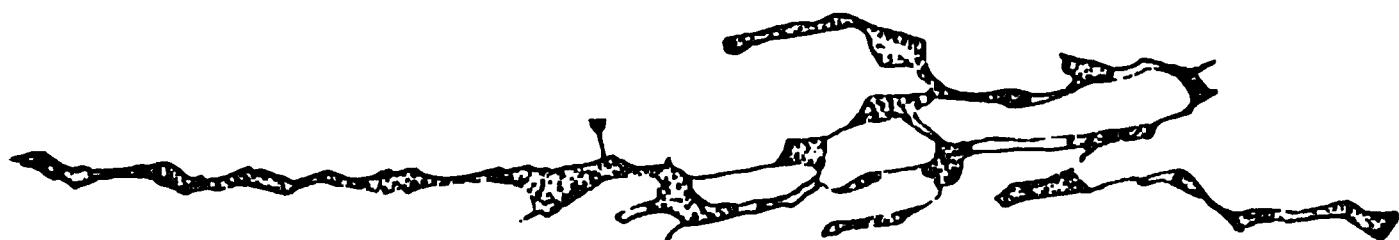


Fig. 4.

carbon dioxide, is 2.28 mms in length. The largest cavities noted by Brewster contained only the dense liquid; the largest observed by Vogelsang in topaz reached only 0.5 mm in one direction. Brewster however found in sapphire a cavity one-third inch in length, two-thirds full of a liquid expanding at  $28^{\circ}$  C. Fine capillary tubes project from many angles of these cavities, often directly toward each other in exact line from adjacent cavities, hinting at a broken connection. Such a relation seems to be indicated by the tubules between the main cavity in Fig. 4, and the shorter one in the lower right-hand part of that figure. The abundance and direction of these tubules plainly show that at the original conditions of temperature during the formation of the topaz crystal, parts of its mass were filled by anastomosing canals occupied by the two fluids, which have been mostly shut up by compression during the ensuing contraction of the mass. That these still remain as imperceptible fissures, was shown by one of Brewster's observations: on heating a small cavity, the liquid was forced along into the apparently compact topaz, through a latent fissure which opened to receive it as it advanced, and then closed up behind it.

The connection or coalescence of straight adjoining cavities at one extremity, frequently results in U-shaped or hook-shaped forms. If

expansions occur at or near the upper ends of the U or hook, and these are occupied by large bubbles of the gas, their contents are naturally in a state of unstable equilibrium, readily subject to movement by changes of temperature in the topaz matrix. This explains the partial transference of contents I have occasionally observed in U-shaped cavities of this kind, portions of the liquid carbon dioxide, or minute bubbles of the gas passing along an arm from one expansion to the other, as in a naturally formed differential thermometer.

In one slice of the topaz the temperature of the disappearance of the gas bubbles was determined on ten cavities, chosen at random, at the following points : 26.6, 27.1, 27.1, 27.1, 27.3, 27.6, 27.6, 27.6, 27.7, 27.8. Though these cavities varied greatly in size, the figures are seen to be very close—within a degree—the gas bubble in the first one noted being proportionately of unusually large size. Several instances of this kind observed incline me to suspect that the wide divergence in the results obtained from cavities in the same slice of rock or mineral, reported by other observers, may have been due, in some cases at least, to the coarse and inexact methods and apparatus hitherto in use.



# THE AMERICAN CHEMICAL SOCIETY.

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## XVI.—PROCEEDINGS.

*Regular meeting, June 3d, 1881.*

The meeting was called to order at 8:30 P.M., Dr. A. R. LEEDS in the chair.

Minutes of previous meeting were read and, after corrections, were approved.

There was no report from the Board of Directors.

The committee appointed to fill the vacancy in the Committee on Papers and Publications, reported that they had selected Dr. ENDEMANN for the position. This recommendation being put to the Society, it was carried unanimously.

The committee appointed to take steps for the drawing up of an obituary notice of Dr. GOLDMARK, reported that Dr. O. H. KRAUSE had been asked to undertake that duty.

The committee appointed to inquire into the question of filling the vacancy in the Board of Directors, caused by Dr. GOLDMARK's death, from a legal point of view had no report at present.

The resignation of Mr. WM. WHEATLEY was then read, and accepted.

Mr. A. P. HALLOCK was then balloted for and duly elected a member of the Society.

After which the following paper was read.

“On the Analysis of Elephants' Milk.” By Dr. CHAS. A. DOREMUS.

In discussing this paper, Dr. LEEDS remarked that the ash closely approximated to that of human milk. He also said, commenting on the difficulty mentioned by Dr. DOREMUS in evaporating the milk, that 10 c.c. of milk upon 30 c.c. of ignited sand worked very well, causing no charring; by employing a large excess of sand the milk does not touch the sides of the dish.

Dr. E. R. SQUIBB then took the chair, and the following paper was read:

“On the Adulteration of Food, Drink and Drugs, from the Chemist's Standpoint, and on the Attitude of Chemists in the matter of the Appointment of Government Analysts.” By Dr. A. R. LEEDS.

Mr. ELLIOTT asked Dr. LEEDS if he knew the composition of the material insoluble in water, which he had found in condensed milk, and Dr. LEEDS replied that he had not yet completed the analysis. To a further inquiry of Mr. ELLIOTT, Dr. LEEDS remarked that he had not made any tests for gelatine in condensed milk.

Dr. DOREMUS remarked that one of the first things to decide in this question of adulteration is, "What is adulteration?" and discussed at some length the matter of alum baking powder, claiming that at the present time there is no proof that they are unwholesome.

Dr. E. R. SQUIBB remarked that he looked upon the matter from a different standpoint than most chemists, and would punish as a fraud anything that was sold and not found as represented. He condemned in strong terms the sale of quack medicines, and remarked that the American people take from five to six times as much medicine, *per capita*, as any other nation.

Mr. EIMER also made some remarks upon the enormous quantities of patent medicines sold in the United States.

On motion, the meeting was adjourned.

ARTHUR H. ELLIOTT,  
*Recording Secretary, pro tem.*

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## XVII.—ON THE COMPOSITION OF ELEPHANTS' MILK.

BY CHARLES A. DOREMUS, M.D., Ph.D.

(Read before the American Chemical Society June 3d, 1881.)

Noticing the recent advertisement in this City regarding the "baby elephant," it occurred to me that perhaps no analysis of the milk of this species of the mammalia had been recorded. This I found corroborated, for though the milk of many animals had been subjected to analysis, no opportunity had ever presented itself to obtain elephants' milk.

Through the courtesy of Mr. Jas. A. Bailey, I was enabled to procure samples of the milk on several occasions.

On March 10th, 1880, the elephant Hebe gave birth to the female calf America. Hebe is now twenty-eight years old, and the father of the calf, Mandrie, thirty-two. Since the birth of the "baby," the mother has been in excellent health except during about ten days, when she suffered from a slight indisposition which soon left her.

When borne the calf weighed  $213\frac{1}{2}$  pounds, and in April, 1881, weighed 900 pounds, a very fair year's growth on a milk diet. At the time I procured the samples, both mother and calf were in fine health.

To obtain the milk was a matter of some difficulty. The calf was constantly sucking; nursing two or three times an hour, morning, noon, and night. The milk could be drawn from either of the two teats, but only in small quantities. The mother gave the fluid freely enough apparently to her infant, but sparingly to inquisitive man, so the ruse had to be resorted to of milking one teat while the calf was at the other.

When I first examined the specimens they seemed watery, but, to my surprise, on allowing the milk to stand, I could not help wondering at the large percentage of cream.

The following represents approximately the daily diet of the mother: 3 pecks of oats, 1 bucket bran mash, 5 or 6 loaves of bread,  $\frac{1}{2}$  bushel of roots (potatoes, etc.), 50 to 75 pounds of hay, and 40 gallons of water.

Elephants eat continually, little at a time, to be sure, but constantly picking. This habit is also observable in the way the calf nurses. The first specimen of milk was procured on the morning of April 5th, the second on the 9th, and the 3d on the 10th.

The last exceeded the others in quantity, and would therefore be the fairest of the three. It took several milkings to get even these, for the calf would begin to nurse, then stop, and when she stopped, the flow of milk did also.

I was assured by Mr. Cross and the keeper, Mr. Copeland, that the milk I obtained had all the appearances of that drawn at various times since the birth of the calf. Mr. Cross, when in Boston, compared the milk with that from an Alderney cow, and found the volume of cream greater.

I endeavored to have the calf kept away from the mother for some hours, but could not, since she is allowed her freedom, worries under restraint, and besides has never been taken from the mother. The calf picked at oats and hay, but was dependent on the mother for nourishment.

It would have been a matter of great satisfaction to me had I been able to obtain a larger quantity of the milk, or to have gained even an approximate knowledge of the daily yield, but was obliged to content myself with what I could get. By comparing several samples, however, a just conclusion regarding the quality was found.

The analyses of the samples gave the following results:

	I. April 5th. MORNING.	II. April 9th. NOON.	III. April 10th. MORNING.
Quantity.....	19 c.c.	36 c.c.	72 c.c.
Cream.....	52.4 vol. %	58	62
Reaction.....	Neutral.	Slightly alkaline.	Slightly acid.
Sp. gr.....	.....	.....	1023.7

## In 100 parts by weight :

Water.....	67.567	69.286	66.697
Solids.....	32.433	30.714	33.303
Fat.....	17.546	19.095	22.070
Solids not fat....	14.887	11.619	11.233
Casein.....	} 14.236	3.694	3.212
Sugar.....		7.267	7.392
Ash.....	0.651	0.658	0.629

Ten grams were taken for analysis, and in No. III duplicates were made. It is evident from these analyses that the milk approaches the composition of cream, yet it did not have the consistency of ordinary cream. A cream even raised upon it. Under the microscope the globules presented a very perfect outline, and were beautifully even in size and very transparent. The cream rose quickly, leaving a layer of bluish tinge below. The milk was pleasant in flavor and odor, and very superior in these respects to that of many animals, such as goats or camels, and quality equal to that of cows. Nor did the milk emit any rank odor on heating.

When 10 grams were evaporated to dryness, the last portions of water were hard to remove, as the residue fairly covered with oil. Only by long continued application of heat, and in analysis III, over sulphuric acid in vacuo, could a constant weight be obtained. I would have used sand in the drying or Baumhauer's method of fat extraction, but for the small quantity of milk at my disposal, and from fear of loss of fat in the latter case.

The fat in III was determined by extracting the dried residue, and also with 20 c.c. of milk, by adding alkali and shaking with ether, removing and evaporating the ether, and weighing the fat.

As is shown in the table, the sp. gr. is very low, though the solids, and solids not fat, are great. The ash, casein and sugar are in about the usual proportion. The weight of casein is, it is true, but half that of the sugar. The milk indeed shows an unusually great preponderance of the non-nitrogenized elements, and this seems to correspond with the wants of the animal, since the fatty tissues are

greatly developed in elephants. According to Mr. Cross, who has had large experience with these animals, they are fatter in the wild state than in bondage. These specimens must appear as exceptional; they may be considered by some as "strippings," but as against such a view we have the recurrence in each sample of the same characteristics in the milk, and a near correspondence in the composition. As may be seen from the subjoined analyses given by v. Gomp-Besauer, the milk belongs to the class of which human and mare's milk are members, especially as regards the proportion of the non-nitrogenized to the nitrogenized elements. It may be remarked that though approaching the composition of cream, it still differs enough to require it to be considered milk.

Perhaps if a larger quantity of the milk could be collected, it would assume a more watery character, and approximate more nearly to other milk in that respect. However this may be, the quality of the fat deserves some attention.

The fat has a light yellow color, resembling olive oil, is very pleasant in odor and taste, is liquid at common temperatures, but solidifies at  $18^{\circ}\text{C.} = 64^{\circ}\text{F.}$

The cow must yield a considerable quantity of milk since the growth of the calf has been constant, and at the time these samples were milked, the mother gave as freely to her babe as she ever had since its birth. The calf having gained 7-800 lbs. on a milk diet in one year, it is presumable that it had no lack of nourishment.

In size the "baby" compared equally with other elephants in the same menagerie, who were known to be four and five years old.

From whatever standpoint, therefore, we view the lacteal product of these four-footed giants, we are fully warranted in ascribing to it not only extreme richness, but also great delicacy of flavor.

## ON THE COMPOSITION OF ELEPHANTS' MILK.

	WOMEN.	COWS.	GOATS.	EWES.	ASSES.	MARES.	BUFFALO COWS.	CAMELS.	SOWS.	HIPPOTAMUS.	ELEPHANTS.
Water .....	86.271	84.28	86.85	83.30	89.01	90.45	80.640	86.34	81.80	90.43	66.697
Solids.....	13.729	15.72	13.52	16.60	10.99	9.55	19.360	13.66	18.20	9.57	33.303
Fat .....	5.370	6.47	4.34	6.05	1.85	1.31	8.450	2.90	6.00	4.51	22.070
Casein .....	2.950	3.57	2.53	5.73	3.57	2.53	4.247	3.67	5.30	4.40	8.212
Albumen.....		0.78	1.26							Milk Sugar included.	
Milk Sugar....	5.136	4.34	3.78	3.96	5.05	5.42	4.518	5.78	6.07		7.392
Ash.....	0.223	0.63	0.65	0.68		0.29	0.845	0.66	0.83	0.11	0.629
Nitrogenized...	2.950	4.35	3.79	5.73	3.57	2.53	4.247	3.67	5.30		3.212
Non-Nitrog'niz'd	10.506	10.81	8.12	10.01	6.90	6.73	12.968	8.68	12.07		29.462

**XVIII.—UPON THE ADULTERATION OF FOOD, DRINK AND DRUGS,  
FROM THE CHEMIST'S STANDPOINT ; AND UPON THE ATTITUDE OF  
CHEMISTS IN THE MATTER OF APPOINTMENT OF "GOVERNMENT  
ANALYSTS."**

BY ALBERT R. LEEDS.

Abstract.

The subject of adulteration had not received that amount of attention from the chemists which its nature and amount, as at present practiced in the United States, properly demanded. And in neglecting to assume control of the investigation of the subject, and of the information and education of the public mind as to the actual facts, they had left the field in the possession of scientific demagogues and pseudo chemists who, for the past four years, had been sending out through the daily press an unlimited amount of sensational literature, either in part or altogether false. The investigations as to the facts had been mainly fragmentary and sporadic in their character, instituted from time to time by various Boards of Health, but without much outcome in the arrest and prosecution of offenders. But this period of education had now gone by, and the time had come when the various States were about to pass laws upon adulteration. Some laws had been already enacted, and grave blunders had been committed. The experience in England had shown how difficult it was to secure wise and efficient legislation, and it was very important for chemists in America to avail themselves of that experience, and to control and shape the laws now under consideration in the legislatures of the various States. Moreover, the duty of the public analyst required public training and accurate knowledge of certain topics not ordinarily studied by chemists. Finally, the analysts could not properly perform their labors without the aid of suitably qualified inspectors, and as yet these two professions did not exist in the country and would have to be in a sense created.

Dr. Leeds, who had been commissioned by the State of New Jersey to investigate the facts concerning adulteration, as a basis for future State legislation, then gave a brief resumé of the results of his labors up to date. He had analysed a very large number of samples, and without claiming that the facts were universally true for the whole country, yet the examinations of so many articles of each kind, purchased at random from dealers in various parts of New Jersey and in the City of New York, might justly be regarded as representing a reasonably close approximation to the true facts in the case.

In regard to teas, three classes of adulterated samples had been found. The smallest class contained an amount of mineral matter in excess of that which should be present from facing materials alone, or from impurities which could properly be derived from the processes of preparation for the market. The largest class consisted of teas adulterated with exhausted leaves. Finally, in some samples foreign leaves were present in such numbers as to denote intentional adulteration. But the percentage of adulterated teas was much smaller than was anticipated (10 per cent.), a majority of the teas being of inferior grade, their prices ranging from thirty to fifty cents per pound. Nearly all the coffees examined, both whole and ground, were pure; the adulterant in the other cases being chiccory. The essences of coffee sometimes contained scarcely any coffee—one sample was composed almost entirely of chiccory, caramel and liquorice.

The manufacture of mixed sugars, containing both cane sugar and glucose, is now largely carried on in the United States, the product being sold under the name of “new process” sugar by the manufacturers. This fact had given rise to a popular belief that almost all varieties of sugar were more or less adulterated. Such was not found to be the case, most of the white sugars, purchased at random, being found pure; and of the brown sugars, which were the more adulterated, certain samples furnished by dealers as in their belief adulterated, were found to contain no more inverted sugar than might properly be present in a sugar of a low grade. A similar remark applies to the sirups. Even the lowest priced sirups were found, with few exceptions, to contain no more glucose than was to be expected in a non-adulterated article. No free sulphuric acid, or excess of lime, was present in any case.

The specimens of flour examined were uniformly pure, no other meal or alum being found in any instance. In bread, however, sufficient samples showing the employment of alum were obtained to demonstrate its occasional use by bakers. Cream of tartar was found to be extensively adulterated, the adulterants being sulphate of lime, acid phosphate of lime and starch. The first-named substance was likewise present in some samples of bicarbonate of soda.

Some of the manufacturers of the baking powders now so extensively sold, use only bicarbonate of soda, cream of tartar and starch. Others employ either partly or altogether, in the place of the cream of tartar, alum or acid phosphate of lime, or a mixture of these two salts. Of the condiments and spices very few were pure, though in no instance was any injurious metallic adulterant detected.



Many of the samples of vinegar contained a much lower percentage of acetic acid than should have been present, but in no case were mineral acids present, and in only one instance a metallic substance—lead in traces. Some of the so-called white wine vinegars had all the characters of ordinary cider vinegar, decolorised by filtration through animal charcoal. None of the green pickles examined were free from copper—one pickle containing 15 milligrams—although the pickles prepared by the use of brine, and those imported, with the Crosse & Blackwell label, were found unexceptionable. The employment of tin cans in the preservation of vegetables is reprehensible, both tin and lead being present in the canned tomatoes examined. The skimming and watering of milk was still very extensively practiced in New York and New Jersey, although stringent laws had been passed for its repression.

Condensed milk varied remarkably in composition, the percentage of water ranging from 25.5 to 59 per cent., and of fat from 3 to 11 per cent.





# THE AMERICAN CHEMICAL SOCIETY.

## Reports on American and Foreign Patents Relating to Chemistry.

### American Patents.

Condensed from the Official Gazette of the U. S. Patent Office, by ARNO BEHR.

*Jan. 4, 1881.*

236,150.—*Lubricating compound.* PATRICK H. FITCH.

Soap, sal-soda, carbonate of ammonia, sal-ammoniac, red lead, naphtha and water.

236,198.—*Method of waterproofing textile fabrics, leather, etc.* PETER H. VANDER WEYDE.

The objects to be waterproofed are placed in a closed box and subjected to the vapors evolved by heating ozokerite to 400 or 500° F.

236,216.—*Marine paint.* ROBERT H. DIMOCK.

Linseed oil, suboxide of copper and carbolic acid.

236,240.—*Treating caoutchouc.* GEORGE M. MOWBRAY.

Caoutchouc combined with naphthalene.

236,280.—*Composition for tanning hides and skins.* HEINRICH TRENK.

Two watery solutions are used. the first of pyroligneous acid and chromate of alumina, the second of crude tartar and chloride of zinc.

236,248.—*Vegetable soup compound.* JOHN D. WARREN.

Vegetables cut and dried, and mixed with salicylic acid and sulphate of soda.

236,299.—*Process of, and apparatus for, evaporating saccharine liquids.* GEORGE B. BOOMER.

236,330.—*Process for the manufacture of glucose.* RUDOLPH D' HEUREUSE.

Refers to a mechanical treatment of the steeped corn, preliminary to the manufacture of the starchy portion into starch or glucose, which treatment has for its object the separation of the hulls and germs, and the removal of the soluble gluten.

The successive use of oxalic acid and soda for the removal of lime from the converted product, is also claimed.

*Jan. 11, 1881.*

236,471.—*Ice-making machine.* FRANZ WINDHAUSEN.

236,480.—*Compound for manufacture of gas tubing.* SAMUEL BARR.

A composition of glue, glycerine, soap, borax, and a solution of sulphate of iron, to be applied between the two textile coverings of the tubing.

236,483.—*Substitute for butter.* OTTO BOYSEN.

A mixture of oleomargarine and an alkaline solution is agitated until partial saponification is effected, and then a minute quantity of butyric acid is added.

236,506.—*Compound filling for fireproof structures, such as safes, chests, bank vaults and doors.* WILLIS B. MARVIN.

Essentially a mixture of anhydrous sulphate of lime and fibrous asbestos.

236,521.—*Preserving compound.* JEAN WICKERSHEIMER.

A mixture of salicylic acid, methylic alcohol, glycerine, and a solution of alum, common salt and potash, for the preservation of meat intended for food.

236,559.—*Tanning Hides.* ROBERT F. and ISAAC DOBSON.

After immersion for several days in a bath of strong brine and tanning extract, the hides are subjected to the action of gaseous sulphurous acid.

236,598.—*Preserving eggs.* CHARLES H. KIRKHAM.

The substance of the egg is, before desiccation, mixed with a small proportion of starch paste.

236,600.—*Process of, and apparatus for, treating animal matter.* ALFRED and EDWIN LISTER.

Refers to the drying of the animal matter.

*Jan. 18, 1881.*

236,709.—*Composition for treating rubber cloth.* PHILIPP KROPP.

Linseed oil, oxide of manganese, copal and coloring matter.

236,714.—*Manufacture of explosive compounds.* CHARLES A. MORSE.

The explosive consists of nitroglycerine, a resinous substance, and nitre intimately mixed.

236,739.—*Process of, and apparatus for, treatment of ores.* THOMAS G. WALKER.

The process consists in blowing powdered ore, together with air, through heated pipes, by means of a steam jet.

236,763.—*Process of manufacturing artificial manure.* FRANCIS J. BOLTON and JAMES A. WANKLYN.

Urine is evaporated to dryness in contact with small proportion of charcoal, or a similar material.

236,764.—*Apparatus for making vinegar.* OSCAR F. BOOMER and HENRY R. RANDALL.

236,778 and 236,779.—*Process of desulphurising and devulcanising waste vulcanised india rubber.* HENRY A. CLARK.

These processes seem to consist in a treatment with vapor of turpentine.

- 236,843.—*Artificial production of ice and cold.* AUGUSTE J. ROSSI and LEONARD F. BECKWITH.

The liquid consists of a solution of ammonia gas in glycerine.

*Jan. 25, 1881.*

- 236,878.—*Apparatus for purifying salt.* JOHN H. DUNCAN.

- 236,940.—*Apparatus for evaporating solutions in contact with air or other gas.* JULES L. FAESCH.

- 236,995.—*Bituminous cement.* EDWARD J. DE SMEDT.

Heats coal-tar with an oxidizing agent.

- 237,007.—*Tanning compound.* JAMES FOLEY.

An ordinary tanning solution mixed with the extract of the wood of *Morus tinctoria*.

- 237,017.—*Paint.* JOHN F. HOFFMANN.

Impure carbolic acid mixed with lime, rosin, and asphaltum.

*Feb. 1, 1881.*

- 237,113.—*Apparatus for recovering soda from waste alkaline liquor.* SAMUEL LEE.

- 237,134.—*Filtering apparatus.* CHARLES H. SENFF and PAUL CASAMAJOR.

A sheet of filtering material is rolled in a number of layers around a perforated drum, in the interior of which a partial vacuum is created. The whole is immersed in the liquor to be filtered. By turning the drum, and withdrawing the cloth, the filtering surface is continually renewed as long as the cloth lasts.

- 237,217.—*Process of extracting gold and silver from their ores.* CHARLES DE VAURIAL.

- 237,249.—*Treatment of vulcanised india rubber and gutta-percha.* HENRY A. CLARK.

Claim: The combination with desulphurized india rubber of a vegetable oil or a resinous substance.

- 237,252.—*Compound for making and treating steel.* JOHN CONANT.

Sulphate of copper, rosin and sal ammonia.

*Feb. 8, 1881.*

- 237,484.—*Process of refining vaseline.* ROBERT A. CHESEBOROUGH.

It is kept just at the point of vaporisation in an open vessel until the smelling portions are driven off, and afterwards filtered through bone-black.

- 237,497.—*Method of making articles from waste amber.* ABBOT R. DAVIS.

The method consists in treating the pieces with solvents and subjecting them to pressure.

237,630.—*Process of preparing hides for tanning.* JAMES S. SWAN.

Subjecting the limed hides to the action of an aqueous solution of alum.

*Feb. 15, 1881.*

237,816.—*Purifying sulphate of alumina.* WILLIAM, THOMAS and JAMES CHADWICK, and JOSIAH W. KYNASTON.

Iron is precipitated with ferrocyanide of calcium, and arsenic dissolved in a sulphide, the latter precipitate carrying down the former.

237,830.—*Galvanic battery.* A. FLOYD DELAFIELD.

237,835.—*Purification of syrups and molasses in the manufacture of sugar from beet-root and cane.* AUGUSTE P. DUBRUNFAUT.

To the liquids to be subjected to the process of osmose, a certain amount of lime is added. When the action of osmose is finished, the lime is removed with carbonic acid.

237,878.—*Fuel.* GEORGE KELLY.

Coal-dust ground with lime, and formed, while in a wet state, into masses of convenient size and shape.

237,905.—*Process of preserving meats.* JOHN L. REESE.

Packing the meat in melted fat charged with sulphurous acid.

237,917 and 237,918.—*Production of oxychinoline.* ZDENKO H. SKRAUP.

It is produced by the action of glycerine and sulphuric acid upon a mixture, either of orthonitrophenol and orthoamidophenol, or of paranitrophenol and paramidophenol.

*Feb. 22, 1881.*

238,133.—*Manufacture of fertilizers.* GEO. T. LEWIS.

Exposes a mixture of an insoluble phosphate and pyrites to the action of atmospheric oxygen and moisture.

*March 1, 1881.*

238,240.—*Fertilizers.* JOSEPH M. and JOHN LIPPINCOTT.

The slag produced in the manufacture of pig iron is proposed as an ingredient of a fertilizer.

238,277.—*Process of obtaining the perfumes of natural flowers by absorption.* ROBERT A. CHESEBOROUGH.

The flowers are steeped in vaseline; the latter taking up the essential oils is transformed into a perfume.

238,389.—*Manufacture of leather.* CHRISTIAN HEINZERLING.

Treating the hides with chromic acid, and afterwards with a solution of stearine.

238,474.—*Treatment of starch and starchy substances, and the production therefrom of a compound body capable of being used as a substitute for malt in brewing, and for other purposes.* CORNELIUS O'SULLIVAN and WILLIAM G. VALENTIN.

Conversion of starch with sulphuric acid, until the dry substance has a specific rotation of 171, and Fehling's solution indicates 44 per cent. of grape sugar in the dry substance.

*March 8, 1881.*

238,509.—*Process for sugar refining, and apparatus for carrying on the same.* S. MORRIS LITTLE.

Uses the sirups separated from high grade sugars for liquoring or washing low grade sugars in a centrifugal machine.

238,570.—*Baking powder.* CHARLES A. CATLIN.

Sulphate of ammonia and bicarbonate of soda.

238,613.—*Manufacture of aluminous cake.* CONRAD SEMPER.

Reduces the iron to the ferrous salt by addition of oxalic acid.

238,680.—*White zinc pigment, and mode of manufacturing the same.* THOMAS GRIFFITHS.

The precipitated mixture of sulphide of zinc and sulphate of barium is mixed with common salt and heated to a bright red heat, and the salt is afterward dissolved out.

*March 15, 1881.*

238,867.—*Petroleum illuminating oil.* HENRY V. P. DRAPER.

Petroleum mixed with a small proportion of chloroform.

238,916.—*Explosive compound.* FREDERICK C. KEIL.

Nitroglucose, nitrate and chlorate of potash, and prepared vegetable fiber.

238,980.—*Manufacture of metallic compounds from sulphur and sulphides.* JOHN B. SPENCE.

*March 22, 1881.*

239,033.—*Preserving wood.* BRANDT V. B. DIXON and JOSEPH P. CARD.

Wood is impregnated with chloride of lead.

239,089.—*Manufacture of sulphate of alumina.* JOS. H. EASTWICK.

The claim is for decomposing a mixture of ground halloysite and hydrate of alumina with sulphuric acid.

239,242.—*Incrustation Preventative.* WILLIAM J. GILLESPIE.

Soda-ash, linseed oil cake, oak bark, copperas, potatoe starch and charcoal.



*March 29, 1881.*

239,346.—*Process of obtaining magnesia.* CARL SCHEIBLER.

Lime is removed from impure burned magnesia by dissolving it in a sugar solution.

239,394.—*Process of, and apparatus for, manufacturing phosphoric anhydride.* H. S. MAXIM.

Claims the bringing together of a jet of vapor of phosphorus, and a continuous blast of air of sufficient volume to oxidise all the phosphorus to phosphoric anhydride.

239,417.—*Incrustation preventative.* PASQUALE ALFIERI.

Carbonate of baryta, nitrate of ammonia, chloride of sodium, and vegetable charcoal.

239,423 to 239,425.—*Treating pyroxyline.* LEONARD S. BEALS.

The object of these patents is the production of a plastic compound containing pyroxyline, nitrobenzole, oil of lavender, benzole, alcohol, wax or paraffine.

*April 5, 1881.*

239,602.—*Baking powder.* CHARLES A. CATLIN.

An alkaline carbonate in combination with sulphate of magnesium.

239,618.—*Process of, and apparatus for, distilling petroleum products.* ALBERT NEILSON.

239,622.—*Art of separating animal fibers from vegetable fibers.* GEORGE B. and ALFRED L. RICE.

The fibers are mixed with bleaching powder, and heated in a close vessel.

239,642.—*Method of preparing anthracite waste for combustion.* ALFRED BERNEY.

The dust is mixed with lime, or soda, and asphalt, or tar, and water.

239,711.—*Manufacture of iron and steel.* ALFRED BRACONNIER.

Superheating the metal, introducing it into a converter, and forcing a current of reducing gas through the molten metal.

239,722.—*Process and apparatus for desiccating substances.* LYDIA J. CADWELL.

*April 12, 1881.*

239,974.—*Indelible ink.* AARON N. MOSES.

Caustic potash, seed-lac, permanganate of potash, and lamp-black.

240,094.—*Petroleum products, and process of obtaining and deodorising the same.* MARTIN CONNELLY.

Heavy petroleum oil is heated with unslaked lime.

240,126.—*Preserving butter.* JOHN HARGER.

A solution of boracic acid in glycerine, and of sulphate of potassium in water, is added either to the cream or butter.

240,196.—*Preparation of hydrochloric acid.* ERNEST SOLVAY.

A solution of chloride of calcium absorbs hydrochloric acid gas, and can be made to give it off again by heating.

*April 19, 1881.*

240,248.—*Process of, and apparatus for, concentrating sulphuric acid.* JUNIUS GRIDLEY.

240,359.—*Manufacture of artificial indigo.* ADOLF BAEYER and HEINRICH CARO.

Xanthate of sodium is used as a reducing agent in order to produce indigo from orthonitrophenylpropionic acid.

240,360.—*Dyeing fabrics with artificial indigo-blue.* ADOLF BAEYER and HEINRICH CARO.

The reaction mentioned in the preceding patent is used to develop the color on the fiber.

240,361.—*Manufacture of artificial indigo.* ADOLF BAEYER.

The dye-stuff produced by the action of ferrous sulphate upon the sulpo-compound of orthonitrophenylpropionic acid is treated with sulphurous acid, and then precipitated with common salt.

240,365.—*Apparatus for testing illuminating fluids.* ALEX. BERNSTEIN.

240,406.—*Filter press.* ALEXANDER GORDEN.

240,493.—*Process of tanning.* GANDENZIO DALLA ZONCA.

*April 26, 1881.*

240,597.—*Manufacture of aluminous cake.* GEORGE T. LEWIS and CARL V. PETRAEUS.

After the removal of the principal portion of the iron by precipitation with yellow prussiate of potash, the rest of the iron is reduced to the ferrous state by zinc.

240,651 and 240,652.—*Treatment of dextrine maltose, and apparatus therefor.* ALEXANDER LOW BRUCE, GEORGE STENHOUSE, WILLIAM McCOWAN and ANDREW HADDOW.

A glucose sirup which the inventors call dextrine maltose, is in thin films brought to a state of dryness by means of specially constructed machines.

240,796.—*Process of, and apparatus for, manufacturing grape sugar.* HORACE WILLIAMS.

The well known practice of artificially cooling the concentrated sugar solution, and of introducing crystallised grape sugar into the mass in order to induce crystallisation, is claimed as new; also a tank provided with cooling pipes, and a stirrer in the shape of a screw.

## Foreign Patents.

Condensed from R. BIEDERMANN'S Report to the German Chemical Society.

by OTTO H. KRAUSE.

G. BORSCHÉ, Leopoldshall: *Method of obtaining potassium-magnesium sulphate from kainite*. (Germ. P., No. 10642, Nov. 4, 1879.)—Crude kainite is dissolved in water of 30–35° C., to a density of 1.25, and crystallized magnesium sulphate added, upon which potassium-magnesium sulphate crystallises out. The mother-liquor is concentrated by evaporation, and mixed with a hot solution of magnesium chloride, whereby nearly all the sodium chloride contained in the kainite is separated as a powder.

R. GRÜNEBERG, Altdamm: *Improved method of working up kainite*. (Germ. P., No. 10754, Dec. 18, 1879.)—Kainite is macerated with a solution of schoenite, saturated in the cold, whereby the kainite is converted into schoenite. Sodium and magnesium chloride enter into solution, and can be used to dissolve native or artificial carnallite.

F. JULLIEN, Lorient: *Manufacture of iodine from marine plants*. (Engl. P., No. 5041, Dec. 9, 1879.)—Marine plants or the varec are systematically extracted, and the lyes precipitated with iron or copper sulphate. The precipitate separated by decantation, is then distilled in retorts.

A. HEGENER, Cologne: *Improvements in apparatus for obtaining ammonia from gas liquors*. (Germ. P., No. 11669, March 11, 1880.)—Two tanks are placed one over the other, and over the gas liquor boiler. The upper one contains milk of lime and is open on top. The lower one is connected with the upper one and with the boiler, by means of cocks and pipes so arranged as to enable steam from the boiler to force the milk of lime received from the upper tank into the gas liquor.

I. P. KAGENBUSCH, Leeds: *Extraction of aluminium and gold from clay*. (Engl. P., No. 4811, Nov. 25, 1879.)—The inventor claims that from clay fused with the aid of fluxes and electrolysis, aluminium can be obtained by cupellation of the alloy of lead and aluminium. Gold is said to be obtained in a similar manner.

OTTO E. POHL, Liverpool: *Manufacture of sulphate*. (Engl. P., No. 5031, Dec. 9, 1879.)—Proposes to make sulphate by bringing sulphurous acid gas and heated air into contact with a solution of sodium chloride which is being evaporated.

AKTIENGESELLSCHAFT FARBWERKE, successors to Meister LUCIUS UND BRUENING, Hoechst, a. M.: *Method of oxidising leuco-bases and their sulphuric acids with the aid of chlorinated quinones*. (Germ. P., No. 11412, Nov. 11, 1879.)—The term leuco-bases is intended to comprise not only leucaniline and the corresponding bases, but also the bases resulting from the combination of aldehydes, or acid chlorides, with primary, secondary and tertiary aromatic monamines. The production of green dyes results chiefly from the oxidation of methylated, ethylated, phenylated and benzylated derivatives of diamidotriphenylmethane and its homologues. Diamidotriphenylmethane gives, by oxidation with chlorinated

quinones, a reddish-blue, not particularly fine, coloring matter. Methylated, ethylated, phenylated and benzylated derivatives of this base give, on the contrary, very fine dyes, some of which are directly soluble in water.

All the leuco-bases from the rosaniline-melt can, by means of chloranile, be converted into coloring matters in the same manner as the leuco-bases of triphenylmethane.

**AKTIENGESSELLSCHAFT FARBWERKE**, successors to Meister LUCIUS UND BRUENING, Hoechst, a. M.: *Improvements in the preparation of coloring matters with the aid of chlorinated quinones.* (Germ. P., No. 11811, Nov. 12, 1879; addition to Germ. P., No. 8251, June 24, 1879.)—By employing the various mono and dimethylated, mono and diethylated bases, dyes are obtained of different shades, from reddish-violet to bluish-violet, which are all soluble in water.

**GEBRUEDER RAMDOHR**, Wanzleben: *Manufacture of magnesia from magnesium chloride, by heating it to redness in the oxidising flame, and passing steam over it.* (Germ. P., No. 11540, Oct. 9, 1879.)—Relates to practical details of the process in which this well known reaction is employed.

**H. GUENTHER**, Berlin: *Improvements in the method of preparing black printing ink.* (Germ. P., No. 11930, May 8, 1880; addition to Germ. P., No. 9566, Oct. 28, 1879, see this JOURNAL, 2, 231.)—Heavy tar oil from the manufacture of anthracene is boiled with 10 per cent. cupric chloride dissolved in water, whereby a saving of aniline violet results.

**M. WEINRICH**, Vienna: *Treatment of sugar solutions.* (Engl. P., No. 4831, Nov. 26, 1879.)—To the heated beet sirup, calcium hydrate and milk of lime are added, to produce saccharate of lime.

**E. MOURLOT**, Paris: *Substitute for gutta-percha.* (Engl. P., No. 4846, Nov. 27, 1879.)—The tar which remains in the retort after the dry distillation of birch bark.

**CHARLES TELLIER**, Paris: *Treatment of coal.* (Engl. P., No. 4473, Nov. 3, 1879.)—The coal is separated from foreign matters by floating it off in a concentrated solution of calcium or magnesium chloride. (A process similar to the above was described by Hargreaves, *Dingler's Polyt. Journ.*, 1868. R. B.)

**JAMES WEBSTER**, Edgbaston: *Preparation of aluminium bronze.* (Germ. P., No. 11577, April 2, 1880.)—Aluminium is deposited electrolytically upon granular or sheet copper to the extent of 1 to 10 per cent., and then combined with 1 to 6 per cent. of an alloy composed of 20 pts. nickel, 20 pts. copper, 30 pts. tin and 7 pts. aluminium.

**A. VON KERPELY**, Chemnitz: *Method of preparing bricks from limestone or dolomite.* (Germ. P., No. 11348, Feb. 10, 1880.)—Burned limestone or dolomite is made into a paste with vinegar, and the resulting highly plastic mass formed into bricks, and calcined at a white heat.

**JACOB OERTLE**, Nuremberg: *Apparatus for producing illuminating gas from gasoline.* (Germ. P., No. 11121, Feb. 28, 1880.)—Two connected pipes filled with wool, in the first of which an upward current of air comes in contact with the gasoline fed in from a vessel above. The second pipe serves as a purifier.

W. HENDERSON, Irvine: *Treatment of certain ores.* (Engl. P., No. 4481, Nov. 3, 1879.)—Ores containing silver, cobalt, nickel and zinc, are roasted, the oxides mixed with acid sodium sulphate, heated to low redness, and extracted with water containing sulphuric acid.

P. G. L. DESIGNOLLE, Paris: *Extraction of noble metals by means of mechanical and electrochemical amalgamation, and apparatus for the same.* (Germ. P., No. 11415, Feb. 8, 1880.)—The ore is worked together with a slightly acid mercurial solution in a rotating cylinder filled with iron balls. When the amalgamation of the gold is complete, the mass is transferred to a second apparatus, consisting essentially of amalgamated copper or silver plates which retain the gold amalgam contained in the mud received from the cylinder.

W. MÜLLER, Antwerp, and E. GEISENBERGER, Brussels: *Apparatus for obtaining ammonia from atmospheric nitrogen.* (Germ. P., No. 11489, June 19, 1879.)—See their Amer. Pat., this JOURNAL, 2, 142.

W. CHADWICK et. al., St. HELENS: *Manufacture of alum and sulphate of alumina free from iron.* (Germ. P., No. 11137, Oct. 10, 1879.)—See their Amer. Pat., this JOURNAL, 2, 440.

E. NEUMANN, Rosswein: *Method for utilising the waste water from wool-washing establishments and fulling mills.* (Germ. P., No. 11112, Dec. 19, 1879.)—The waters are treated in pits with milk of lime, the lime soap thereby formed precipitates the greater part of the impurities. Glue or other nitrogenous matters may subsequently be removed by means of a weak solution of tannin. The deposit of insoluble salts of the fatty acid is subjected to destructive distillation to obtain ammonia, etc.

FERD. CAMUS, Paris: *Treatment of corn and other cereals.* (Germ. P., No. 11501, March 12, 1880.)—The grains are bleached by soaking them in a solution of acid sodium, or calcium sulphite, and adding an acid.

G. F. MEYER, Braunschweig: *Method of filtration of beet juice.* (Germ. P., No. 11296, Feb. 13, 1880.)—Besides sand, mineral, wool, etc., mentioned in his first patent (see this JOURNAL, 1, 305), the inventor now claims also the use of quartz, coal, coke, and the insoluble compounds of the alkaline earths, as substitutes for bone-black.

P. H. H. NEUMANN, Brussels: *Method of rendering parchment paper opaque and pliable.* (Germ. P., No. 10008, Jan. 8, 1880.)—The property mentioned is given to it by adding baryta salts to the pulp, or applying them in the solution to the paper before the treatment with sulphuric acid, the latter by adding calcium chloride or other hygroscopic substance.

H. GUETTLER, Kriewald: *Use of dextrine in the preparation of cartridges from compressed blasting powder.* (Germ. P., No. 10978, Dec. 3, 1879.)—The mixture is impregnated with solution of dextrine and the plastic mass compressed. A coating of shellac renders it waterproof.

T. H. COBLEY and W. G. GARD, Dunstable: *Method of preparing tannin black for printing ink, blacking, etc.* (Number and date of patent not given.)—Leather waste and animal matters containing glue or gelatine, and materials containing tannin, are extracted with alkaline water. The solution is then precipitated with an iron salt, and the precipitate mixed with tar oil.

CHARLES WIGG, Liverpool: *Manufacture of potassium and sodium sulphate.* (Engl. P., No. 5105, Dec. 18, 1879.)—Superheated steam is injected into the sulphate furnace. The hydrochloric acid gas evolved being free from air, can be more readily condensed.

HENRY BRUNNER, Widnes: *Manufacture of soda.* (Engl. P., No. 5228, Dec. 22, 1879.)—The inventor adds sodium nitrate, or the oxides or carbonates of zinc, lead, iron and manganese, to the lyes to oxidise or decompose sulphides, and to remove silicate of alumina, etc.

G. BORSCHÉ, Leopoldshall: *Improvements in the method of obtaining potassium-magnesium sulphate and carnallite from kainite.* (Germ. P., No. 11028, Dec. 4, 1879; addition to Germ. P., No. 10642, see this JOURNAL, 3, 70.)—Sodium sulphate may be employed instead of magnesium sulphate for the purpose of precipitating the potassium-magnesium sulphate.

R. POWELL, Liverpool: *Manufacture of calcium sulphite.* (Engl. P., No. 5109, Dec. 13, 1879.)—Dry process. Sulphurous acid is brought into contact with pulverised lime, or with calcium carbonate, in a series of lead chambers.

GEBRÜEDER RAMDOHR, Wanzleben: *Manufacture of magnesia from magnesium chloride.* (Germ. P., No. 11746, March 7, 1880; addition to Germ. P., No. 11580, Oct. 9, 1879.)—The solution of magnesium chloride is evaporated by direct application of an oxidising flame, and heated in presence of superheated steam to drive off a large part of the hydrochloric acid. The residual mixture of MgO and MgCl<sub>2</sub> is mixed with iron ore or clay, and formed into bricks which are burned at a high temperature in presence of steam.

H. UELSMANN, Koenigshuette: *Use of silicious iron in galvanic batteries.* (Germ. P., No. 11284, May 11, 1880.)—The iron in a zinc-iron battery is replaced by pig iron containing 12 per cent. of silicium.

JAMES MACTEAR, Glasgow: *Preparation of crystalline carbon.* (Engl. P., No. 5143, Dec. 16, 1879.)—The inventor proposes to prepare carbon in a diamond-like form by subjecting carbon compounds to a very high pressure and high temperature, preferably in presence of a body capable of uniting with the elements combined with the carbon in such compounds.

JAMES MACTEAR, Glasgow: *Preparation of crystallised carbon.* (Engl. P., No. 45, Jan. 6, 1880.)—Carbonic acid and finely divided iron, or oxalic acid and oxidisable metals, are heated for 70 hours in steel vessels to a temperature of 300 to 400° C. The finely crystallised carbon so obtained, is intended to be used as a substitute for emery.

A. BAEYER, Munich: *Preparation of the derivatives of orthonitrocinnamic acid, the homologues and substitution compounds of these derivatives, and conversion of the same into indigo-blue and analogous coloring matters.* (Germ. P., No. 11857, March 19, 1880.)

A. BAEYER, Munich: *Improvements in the methods of preparing the materials used in the manufacture of indigo, and method for the direct production of this coloring matter upon the fiber.* (Germ. P., No. 11858, March 19, 1880; addition to Germ. P., No. 11857.)—See Prof. Baeyer's American patents, this JOURNAL, 2, 228, 295, 403 and 457.

A. ENGELER, Winterthur: *Dry method of bleaching cotton by the use of chlorine-form vapors containing chlorine.* (Germ. P., No. 12127, June 26, 1880.)—The bleaching gas is prepared by allowing sulphuric acid to react upon one part unslaked lime, one part chloride of lime, and one part alcohol.

ORAZIO LUGO, Flushing, and W. T. LEES, Brooklyn: *Improvements in the purification of gas.* (Germ. P., No. 11247, Dec. 19, 1879.)—The crude gas is mixed, between the condenser and the scrubber, with 1 to 2 per cent. of air, and the mixture heated to 250° by means of superheated steam.

A. NOBEL, Paris: *Application of compressed gunpowder in combination with fulminating cartridges composed of dynamite, or of a mixture of gunpowder and dynamite, nitrogelatine, nitrocellulose, bipicrate of baryta, lead or potassa, for blasting purposes.* (Germ. P., No. 11030, Jan. 6, 1880.)—The object of this invention is to render compressed, ungrained gunpowder serviceable for blasting.

V. HAENING, Dresden, and O. REINHARD, Loschwitz: *Extractor with return condenser.* (Germ. P., No. 10771, Feb. 10, 1880; addition to Germ. P., No. 6737.)—The apparatus is composed of two parts. The upper one contains the matter to be extracted, the lower one the extracting liquid. The cover forms the return condenser, being so shaped as to hold the cooling water. Immediately below this, and connected with it, is the cooling pipe. By means of a branch pipe direct steam can be admitted to the upper compartment, whence it passes as extract into the lower one. As soon as sufficient liquid has collected here, the steam by the branch pipe is shut off, and the process of extracting carried on in the usual way.

I. BALLATSCHANO, C. BALLATSCHANO and H. TRENK, Berlin: *Method of tanning.* (Germ. P., No. 11031, Jan. 10, 1880.)—The following three baths are employed: 1. A solution of chromate of alumina in wood alcohol; 2. A concentrated solution of cream of tartar and nickel-ammonium chloride; 3. A solution of 1 to 2 parts tannin in 1,000 pts. water, and 20 to 25 parts of wood alcohol.

G. PRAETORIUS, Breslau: *Method of making porous objects and vessels of earthenware to be used for filtration, absorption and dialysis.* (Germ. P., No. 11810, Oct. 9, 1879.)—Charcoal powder is added to the clay to produce porosity.

R. A. FISHER, Philadelphia: *Neutral combination of aluminium and zinc sulphate for paper manufacture.* (Engl. P., No. 170, Jan. 14, 1880.)—Oxide of zinc is dissolved in a solution of aluminium sulphate until it is so neutral as not to change coloring matters.

H. GERNER, New York: *Treatment of caoutchouc, gutta-percha, etc.* (Engl. P., No. 47, Jan. 6, 1880.)—Camphor dissolved in alcohol or benzole is incorporated with the mass before vulcanising.

I. A. W. WOLTERS, Dresden: *Method of preparing sulphuric anhydride.* (Germ. P., Jan. 7, 1880.)—Alkaline pyrosulphates are decomposed with sulphuric acid.



The acid sulphate remaining after the distillation is reconverted into pyrosulphate by heating.



Mr. HELLOFF, Berlin: *Blasting compounds*. (Germ. P., No. 12122, June 3, 1880.)—Crude tar oil is treated with concentrated nitric acid. The nitro-compounds formed are mixed with chlorates or nitrates, or with nitric acid.

B. BERNHARDI, Stassfurt: *Combined method for the manufacture of schoenite, potassium sulphate and chloride*. (Germ. P., No. 12498, April 13, 1880.)—Relates to the employment of lyes resulting from the manufacture of schoenite, or potassium sulphate, instead of water, in the preparation of potassium salts.

R. S. NEWALL, Gateshead upon Tyne: *Calcining furnace*. (Engl. P., No. 5230, Dec. 22, 1879.)—Improvement upon Mactear's rotating furnace.

I. H. WILHELM, Homburg, v. d. H., and F. BOHNSTADT, Frankfort, a. M.: *Manufacture of printing ink from coal tar*. (Germ. P., No. 12282, Dec. 25, 1879.)—Coal tar is mixed with 6 to 15 per cent. of rosin and 10 per cent. of paraffine oil, and strained. After deodorising with chloride of lime, or potassa, 20 to 25 per cent. of glycerine and 12 to 18 per cent. of lamp-black are added to it. According to an additional patent (Germ. P., No. 12286, Jan. 9, 1880), the tar is first treated with  $2\frac{1}{4}$  to 3 per cent. of sulphuric acid, neutralised with soda, and deodorised with chlorine as above. Finally lard, glycerine, soap, etc., are added.

M. SALZMANN and F. KRÜGER, Fürstenberg, a. O.: *Preparation of red, violet and green coloring matters by the action of chloropicrine upon aromatic amines*. (Germ. P., No. 12096, March 17, 1880.)—Aniline and its homologues are heated with chloropicrine at  $110^{\circ}$ – $120^{\circ}$  C. for some time. Red coloring matters, soluble in water, and violet ones, soluble in alcohol, are produced. The part soluble in water is precipitated with salt; the insoluble part is converted into the sulphonic acid. Instead of the bases their salts may be employed, and the reaction may take place in the presence of metallic salts.

O. KOHLBAUSCH, Vienna: *Method of obtaining tannic acid and extracts of dye-woods by means of dialysis*. (Germ. P., No. 12296, Dec. 11, 1880.)—Instead of rasping the wood, it is cut into thin chips and treated with hot water in a diffusion battery which is similar to those employed for extracting sugar from beets.

I. P. CLOSSON, Paris: *Improvements in the process of obtaining magnesia from dolomite*. (Germ. P., No. 12456, Oct. 23, 1879.)—A solution of calcium and magnesium chloride is prepared by decomposing calcined dolomite with manganoous chloride from the manufacture of chlorine, or with ferrous chloride (from pyrites), or with ammonium chloride from the ammonia-soda process, etc.; hydroxide of manganese, hydroxide of iron and ammonia being respectively formed. The solution of magnesium and calcium chloride is treated with additional quantities of calcined dolomite powder to precipitate the magnesia.

T. F. WILKINS, Clapham: *Preservation of fish*. (Engl. P., No. 501, Feb. 4, 1880.)—Treatment with a solution of metaphosphoric acid and sugar.

I. MACGAAN, Liverpool: *Manufacture of spirits and fermented liquors*. (Engl. P., No. 512, Feb. 5, 1880.)—Glucose prepared by digesting Indian meal with sulphuric acid is subjected to fermentation. The following salts are added to serve as nutriment for the yeast: Acid potassium phosphate (1.3 pts.), potassium carbonate (0.86 pts.), potassium silicate (0.04 pts.), ammonium tartrate (1.50 pts.), ferrous sulphate (0.17 pts.)

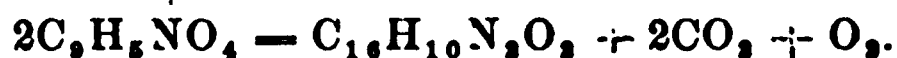


C. C. WALKER, Sallop: *Purification of coal gas*. (Engl. P., No. 545, Feb. 7, 1880.)—Before passing to the scrubber the gas is led through one or more small towers containing a number of metallic screens which are intended to retain any tar carried over.

T. A. EDISON, Menlo Park: *Anaesthetic*. (Engl. P., No. 599, Feb. 11, 1880.)—Composed of chloral hydrate, alcohol, chloroform, camphor, oil of peppermint, oil of cloves, salicylic acid, amyl nitrate, morphine sulphate and ether.

JOS. A. LANFREY and J. L. RENARD, Chartres: *Improvements in the preparation of nitrocellulose paper*. (Germ. P., No. 12115, Jan. 21, 1880.)—The object of this invention is the manufacture of an explosive compound composed essentially of nitrocellulose from straw. This nitrocellulose can absorb 75 per cent. of nitroglycerine.

ADOLPH BAEYER, Munich: *Manufacture of artificial indigo and analogous coloring matters from orthonitrocinnamic acid, and its homologues and substitution products*. (Germ. P., No. 12601, June 18, 1880; second additional patent to No. 11857, of March 19, 1880.)—From the above mentioned substances, orthonitrophenylpropionic acid, its homologues or substitution products are prepared; these are dissolved in cold concentrated sulphuric acid, and treated with sulphate of iron. In consequence of the reducing action of the sulphate of iron, indigo blue, or homologues and substitution products of it, are formed according to the following equation:



H. F. PEASE, W. JONES and JOHN WALSH, Middlesborough, o. T.: *Improvements in rotating furnaces and pans used in the manufacture of potassium and sodium sulphate*. (Germ. P., No. 11953, April 18, 1880; addition to Germ. P., No. 1125, Oct. 20, 1877.)—The improvements relate to stirring and discharging arrangements in a rotating sulphate furnace.

I. B. FREEMAN, London: *Manufacture of zinc-white*. (Engl. P., No. 5121, Dec. 13, 1879.)—In order to give the zinc-white more body, the inventor crushes it under extra heavy mill-stones.

OTTO SANDER, Beuel, near Bonn: *Manufacture of silicic acid from furnace slag, and use of the same for purifying oils*. (Germ. P., No. 11951, April 3, 1880.)—Silicic acid obtained by decomposing the slag with sulphuric or hydrochloric acid is boiled with, or used for, filtering dark mineral oils after their treatment with caustic lye and sulphuric acid.

C. VON BUCH, Oxford: *Crystallisation of carbon*. (Engl. P., No. 804, Feb. 24, 1880.)—Vapor of carbon disulphide is subjected, under pressure in a heated glass tube from which the air has been removed, to a current from a Leclanché battery for several weeks. More vapor is forced into the tube from time to time. Transparent crystals are deposited which yielded carbonic acid by combustion in oxygen.

According to a second method, aluminium fluoride is heated to whiteness in a crucible with ammonium carbonate or other carbon compound. The fluorine is said to combine with the carbon, and to yield it again in the crystalline form.

A third process (Engl. P., No. 830, Feb. 29, 1880) is based upon the property which gases, like carbonic acid, possess under high pressure, of dissolving

carbon and separating it in a crystalline form under the influence of electric discharges, or diminished pressure.

JOHN SATTISON, Nevada City: *Method of controlling the explosion of blasting compounds containing potassium chlorate.* (Engl. P., No. 810, Feb. 24, 1880.)—Coarsely pulverised mustard seed, or linseed, is added to the mass to prevent the formation of hard cakes which, by rubbing against each other, often cause spontaneous explosion of the cartridges.

H. DESPECHER, Paris: *Apparatus for distilling substances containing hydrogen.* (Germ. P., No. 9451, Dec. 10, 1878.)—The method relates to the distillation of coal or other substances containing hydrogen and carbon. A limited amount of highly heated air, as rich in nitrogen as possible, being admitted to the furnace, the hydrogen is said to combine, and to form water and ammonia.

B. THUEMMLER and F. E. SEIDEL, Dresden: *Improvements in the treatment of fibers for the purpose of rendering them suitable for spinning.* (Germ. P., No. 11729, Jan. 21, 1880.)—The stalks of nettles, flax, hemp, jute, etc., are crushed, steamed, then treated with milk of lime and caustic soda. Gum, mucilaginous matters, etc., having thereby been removed, the fibers are subjected to bleaching agents. They are then successively treated with an alkaline carbonate, sulphurous acid, soap water and glycerine vapor.

L. MCINTYRE, Glasgow: *Treatment of fish and fish waste for the purpose of utilising the solid, liquid and gaseous products from them, and apparatus therefor.* (Germ. P., No. 12349, Jan. 1, 1880.)—The material is heated in a rotating cylinder surrounded by a steam jacket. The vapors evolved are passed into sulphuric acid to absorb the ammonia. From the liquid matters which the waste yields, oil is obtained. The dry matters are valuable as manure, or they may be used in the manufacture of murexid, on account of the uric acid contained in them.

H. V. HAIG, Cameron Bridge: *Manufacture of yeast.* (Engl. P., No. 5106, Dec. 13, 1879.)

JUL. STEUER, Blasewitz: *Manufacture of artificial mill-stones, grind-stones and rollers.* (Germ. P., No. 11507, March 31, 1880.)—20 to 70 pts. quartz sand, 70 to 20 pts. porphyry and 5 pts. feldspar, calcareous spar, or fluor spar, are finely ground, mixed with 5 pts. solution of silicate of soda, formed in moulds, and calcined.

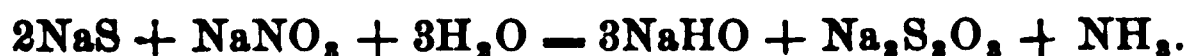
L. WOLLHEIM, Vienna: *Apparatus for collecting non-metallic solid products of electrolytic decomposition.* (Germ. P., No. 11737, May 25, 1880.)—The plate-shaped electrodes are contained in a trough which is so arranged that the liquid to be decomposed enters continuously at the upper end, whilst the products of decomposition pass out through channels near the bottom.

W. KNAUER, Osmuende: *Purification of waste waters from sugar works and other industrial establishments.* (Germ. P., No. 12552, Aug. 1, 1880.)—Instead of purifying the waters, as described in a previous patent, the inventor proposes to allow them to flow over a surface cooler, arranged in the form of a terrace placed under roof.

SACHSENROEDER and GOTTFRIED, Leipsic: *Liquid refined sugar.* (Germ. P., No. 11964, May 23, 1880.)—Dissolved sugar is evaporated with citric or tartaric acid, after which the acid is neutralised with bicarbonate of soda.

H. DRENKEMANN and M. MEIXNER, Basle: *Preparation of pencils for glass painting.* (Germ. P., No. 12662, May 23, 1880.)—Metallic colors are formed into pencils by means of a mixture of 2 pts. stearine oil and 6 pts. stearic acid.

E. CAREY, H. GASKELL and J. HURTER, Widnes: *Purification of alkaline solutions, and generation of ammonia.* (Engl. P., No. 608, Feb. 11, 1880.)—The alkaline solution is first treated with carbonic acid to precipitate alumina and silicic acid, after which the sulphides are oxidised by means of manganic oxide or sodium nitrate. Ammonia is generated when sodium nitrate is employed for oxidising the heated solution—



H. and E. ALBERT, Biebrich: *Extraction of difficultly soluble phosphoric acids from calcic phosphates which have been treated with sulphuric acid.* (Germ. P., No. 12501, May 19, 1880.)—About one hour after the phosphates have been mixed with the acid the mixture is passed through a wet mill. With the aid of a filter press all the phosphoric acid not combined with iron or alumina can be obtained.

JULIAN DENBY, Huelva: *Dephosphorising iron.* (Engl. P., No. 690, Feb. 15, 1880.)—Carbonic oxide or ammonia are blown together with the air, or before or afterwards, through the molten iron contained in a Bessemer converter.

H. UELSMANN, Koenigshuette: *Use of ferrosilicon for apparatus and vessels to contain acids.* (Germ. P., No. 12464, June 15, 1880.)—Iron containing 12 per cent. or more, of silicon is acid proof (see also this JOURNAL, 3, 73).

C. W. SIEMENS, London: *Process and apparatus for compressing molten metals and other substances.* (Germ. P., No. 12037, June 29, 1880.)—Volatile substances such as water, ammonium carbonate, mixtures of nitrates and carbon, are by means of the cover, introduced into the mould containing the liquid steel. The vapors from the substances volatilized by the heat of the metal exert a pressure upon the contents of the mould.

JOHN BENBOW and H. W. SHEPARD, Brooklyn, and R. SEAMAN, New York: *Improvements in the method of coating iron with other metals.* (Engl. P., date and number not given.)—Organic chlorides are employed in conjunction with the metallic chlorides usually employed in galvanising sheet iron.

COUNT DU CHASTENET D'ESTERRE, Paris: *Gilding metals.* (Engl. P., No. 709, Feb. 17, 1880.)—The objects to be gilded are coated with an enamel composed of alumina, cobalt, red lead, boracic acid and powdered glass, and baked. They are then brushed over with the gilding solution, containing gold, tin, arsenious acid, sulphur dissolved in oil of turpentine and oil of lavender, whereupon they are baked.

S. WALKER, Birmingham: *Use of cryolith and sugar of lead in melting copper and obtaining it in compact and even castings.* (Germ. P., No. 12576, April 2, 1880.)—The above mentioned substances are employed alone or together with borax.

W. H. HOOPES, Baltimore: *Manufacture of artificial stone.* (Engl. P., No. 741, Feb. 19, 1880 ; Germ. P., No. 11783, Feb. 26, 1880.)

GEBRUEDER SCHENK, Heidelberg: *Plastic mass*. (Engl. P., No. 650, Feb. 14, 1880.)—Called "tripolith," a substitute for plaster of Paris, over which it is said to have advantages.

J. C. BLOOMFIELD, Castle Cardwell, Ireland: *White cement*. (Engl. P., No. 738, Feb. 19, 1880.)—Lime mixed with 5 per cent. plaster of Paris and with an equal weight pulverised quartz or flint.

G. T. LEWIS, Philadelphia: *Improvements in the manufacture of white lead*. (Engl. P., No. 730, Feb. 18, 1880.)—The lead smoke is blown into a clay retort or muffle heated from the outside so that mixing with the gases from the fire is avoided.

F. G. KROMSCHROEDER, London: *Improvements in the manufacture of illuminating gas*. (Engl. P., No. 619, Feb. 12, 1880.)—Relates to a process of carburetting the gas by passing it through sponges, etc., saturated with hydrocarbons. Excess of the latter is removed by allowing the gas to traverse a compartment of the apparatus in which strips of flannel soaked with acetic acid are suspended.

R. COMBERT, Paris: *Apparatus for purifying oils and fats*. (Germ. P., No. 11460, March 13, 1880.)—The oil is injected by means of steam into the vessel containing the purifying liquid.

J. B. HANNAY, Glasgow: *Antiseptic*. (Engl. P., number not stated.)—Intended for protecting ships' bottoms. It is composed of 45 pts. naphtha, 32 pts. rosin, 12 pts. turpentine and 10 pts. linseed oil, to which arsenite, or chromate of mercury may be added.

JOSEPH DUKE, Totnes: *Fertiliser*. (Engl. P., No. 748, Jan. 20, 1880.)—Gas liquor, sewage, etc., is filtered through a mixture of soluble silicates, superphosphate and peat coke, which retain the nitrogenous matters.

H. W. VOGEL, Berlin: *Improvements in the preparation of photographic emulsions*. (Germ. P., No. 12416, May 8, 1880.)—Four processes are given for combining silver bromide and gelatine emulsions with pyroxyline. The emulsions may be used in the dry or wet condition.

S. W. RIEBS, Croydon: *Hair dye*. (Engl. P., No. 5049, Dec. 9, 1879.)—Manganous acetate and pyrogallie acid with addition of glycerine.

E. S. DAUZIVILLÉ, Paris: *Conversion of wood pulp into glucose and alcohol, and apparatus for same*. (Germ. P., No. 11836, March 23, 1880.)—Wood pulp moistened with hydrochloric acid is exposed in acid proof cylinders to the action of hydrochloric acid gas, and then systematically extracted.

C. PIEPER, Berlin: *Method of preparing saccharate of lime from sirup*. (Germ. P., No. 11342, Oct. 14, 1879.)—Powdered caustic lime and powdered "sirup lime," together with sirup, are passed continuously into a mixing apparatus which can be heated. The stiff paste leaving this apparatus upon an endless belt is cooled and dried in a current of air, and coarsely powdered in a disintegrator.



# THE AMERICAN CHEMICAL SOCIETY.

## XIX.—PROCEEDINGS.

*Regular Meeting, Friday, September 2d, 1881.*

The meeting was called to order, at 8:45 P. M., Dr. A. R. LEEDS in the chair.

The minutes of the previous meeting were read, and approved.

As there had been no meeting of the Board of Directors, the Chair then called upon the Committee on Papers and Publications to report.

Dr. ENDEMANN, in reference to the proceedings, said that all the matter is now in type, and that he expected the JOURNAL would be out in a few days.

The Librarian then reported that the collection of old books, formerly belonging to Prof. TROOSTE, and presented to the Society by Dr. LINDSLY, contains :

	WORKS.	VOLUMES.
Pharmaceutical . . . . .	7	10
Physics and chemistry . . . . .	13	30
Special subjects, phys. and chem. . . . .	6	6
Metallurgy . . . . .	26	29
	—	—
	52	75

As there were no gentlemen to be elected, the next in order was the nomination of new members. The following gentlemen were nominated :

As members :

ALBERT L. COLBY, Ph.B.

Proposed by { ARTHUR H. ELLIOTT,  
ELWYN WALLER,  
ALBERT R. LEEDS.

Dr. HARRY VON BAUER.

Proposed by { H. ENDEMANN,  
JAMES H. STEBBINS, JR.,  
P. CASAMAJOR.

Dr. P. RADENHAUSEN.

Proposed by { H. ENDEMANN,  
JAMES H. STEBBINS, JR.,  
P. CASAMAJOR.

Dr. LOUIS HABEL.

Proposed by { H. ENDEMANN,  
J. F. BATTERSHALL,  
M. BENJAMIN.

H. C. HIEPE.

Proposed by { H. ENDEMANN,  
JAMES H. STEBBINS, JR.,  
J. F. BATTERSHALL.

WM. L. LEMAN.

Proposed by { T. J. PARKER,  
P. CASAMAJOR,  
E. WALLER.

The resignation of Mr. WM. D. MASON was then read and accepted.

The first paper of the evening, "On the Detection of Oleomargarine," by Mr. P. CASAMAJOR was then read by the author.

The detection of this substance is based upon the specific gravity of fats in alcohol, of about 54 per cent., oleomargarine floating in it, whereas the butter sinks to the bottom.

The second paper, "On the Detection of Starch Sugar Syrup, mixed with Sugar-house Syrup," by Mr. P. CASAMAJOR, then followed.

This test depends upon the solubility of sugar-house syrup, in methyl alcohol, while the starch sugar syrup is thrown down as a precipitate. For 1 vol. of syrup it requires about 3 vols. of alcohol, to complete the separation.

The third paper, "On a short Table for testing Sugar by Inversion," by Mr. P. CASAMAJOR, was then read.

The fourth paper, "On a new form of Apparatus for Gas Analysis," by Mr. ARTHUR H. ELLIOTT, then followed. With this apparatus the large amount of washing (as in the case with the Wilkinson apparatus) is done away with. Mr. ELLIOTT found that this washing reduced the illuminants as much as two per cent.

Some remarks by Dr. ENDEMANN and Mr. STEBBINS then followed.

The meeting was then adjourned.

JAMES H. STEBBINS, JR.,  
*Recording Secretary.*

## XX.—DETECTION OF OLEOMARGARINE.

BY P. CASAMAJOR.

In the *Moniteur Scientifique* for April, 1881, is an article on Butter Analysis, in which are given the processes used at the Municipal Laboratory, attached to the Prefecture of Police in Paris, for the detection of foreign fats in butter. This is followed by an account of an arcometric method used for the same purpose, and based on the difference of density between butter and the fats with low melting point, extracted from tallow, which are made to resemble genuine butter, and which are known under the commercial name of *oleomargarine*.

The sale of oleomargarine has become so extensive in this country, that a purchaser of butter is never sure whether he is getting true butter or its imitation. In view of this fact, I have thought it useful to give a process, based on the difference of specific gravity between butter and oleomargarine, of such simplicity that it can be easily applied by any person having rudimentary ideas of manipulation.

Processes of this character are those which can be used with greatest efficiency to check adulterations. I have, in previous communications, given such processes for the detection of starch sugar mixed with cane sugar, and for the detection of starch sugar syrup, mixed with sugar-house syrup.

Although my concern is principally with the difference of density between butter and oleomargarine, I propose to very briefly call attention to the processes used at the Municipal Laboratory of the Prefecture of Police, as these show important differences in chemical composition between true butter and its adulterant, which confirm the difference in the specific gravity. Such an important character as the specific gravity would not differ to any marked extent, without a corresponding diversity in the composition of the two substances.

One process used at the Municipal Laboratory is the following: The sample of butter to be tested is melted, so as to separate water, salt, etc., which is deposited, and a certain amount of scum, which comes to the surface. Of the clear melted fat, under the scum, about 3 or 4 grms. are taken and saponified by 1 or 2 grms. of potassic hydrate. The fat and potassa should be mixed with 50 c.c. of alcohol. In about 5 minutes the saponification is complete, and the cautious addition of water should not produce any turbidity. If any takes place, the operation must be begun anew. The soap formed is afterwards decomposed with weak sulphuric acid, and the insoluble



fat acids are collected and weighed. The result of a great number of experiments is, that in butter the percentage of fat acids thus obtained is usually 86.5 to 87.5 per cent., and that sometimes it is as high as 88 per cent. In animal fats from tallow the percentage of insoluble fat acids is  $95\frac{1}{2}$ . The difference,  $95\frac{1}{2} - 87\frac{1}{2} = 8$  per cent., is attributed to the absence in tallow of volatile and soluble fat acids which exist in butter.

Another process is given in which the result is obtained volumetrically, by estimating the quantity of potassa used in saponifying the fat. 1 grm. of butter requires 225 to 232.4 c.c. of potassa solution, while 1 grm. of tallow, or other animal fat of the same nature, requires from 195 to 197 grms. of the same potassa solution.

Mr. Charles Girard, director of the Municipal Laboratory, considers as adulterated any butter requiring, for saponification, less than 221.5 c.c. of the potassa solution. In some unfavorable cases this volume may represent nearly 30 per cent. of foreign fat.

The method for detecting the difference between butter and oleomargarine by the difference of specific gravity, is one proposed by Messrs. Leune and Harburet.

The butter to be tested is first melted, so as to separate the pure fat from water, salt, etc. The clear melted fat is placed in a cylinder, heated by the vapor escaping from a water bath, kept boiling, but no part of the cylinder is to be in the boiling water. I understand that by heating in this way, the temperature of the melted fat remains at about  $93^{\circ}$  C. To determine the density of this fat an areometer is placed in it. This areometer is graduated in such a way that, in butter, it will sink to the lowest mark of the scale, while oleomargarine corresponds to the highest point in the graduation. The intervening space is divided into ten equal parts, each one of which corresponds to  $\frac{1}{10}$  of oleomargarine, mixed with butter. More than 600 experiments made by Messrs. Leune and Harburet, with artificial mixtures, show that, within an approximation of ten per cent., the instrument gives correct results.

Soon after this areometric method was published, it was announced that the difference of the specific gravities of butter and of oleomargarine, was too slight to distinguish the one from the other. As Messrs. Leune and Harburet had not stated what the specific gravity of each was, it was impossible to judge of the truth of this statement, and it became interesting to ascertain the facts of the case. The following process is the result of my attempts to deter-

mine the specific gravities of butter and of oleomargarine : I chose, in the first place, to ascertain the specific gravity of each at  $15^{\circ}$  C., which is the usual temperature for such determinations. The process consisted in finding for each a liquid in which, at  $15^{\circ}$  C., a portion of butter or of oleomargarine, freed from impurities by previous melting, and containing no air bubbles, would remain in equilibrium in any portion of the liquid, without any tendency to rise to the top or sink to the bottom. The readiest liquid for this purpose was a mixture of alcohol and water, as this is easily prepared and it has no dissolving action on the fats to be tested. As the density of the liquid in which a body remains in equilibrium is the density of the body itself, the problem was narrowed down to finding the difference of density between two mixtures of alcohol and water of different strengths. It was found that pure butter, at  $15^{\circ}$  C., would remain in equilibrium in alcohol of 53.7 per cent. This corresponds to specific gravity 0.926. This butter was obtained from a gentleman at whose country place the butter was made. I obtained oleomargarine from melted warm beef suet by pressure. At a temperature of  $25^{\circ}$  C., this expressed fat had the consistency of butter. The alcohol, which at  $15^{\circ}$  C. would hold it in equilibrium, had a strength of 59.2 per cent., which corresponds to a specific gravity of 0.915.

The question of the possibility of distinguishing butter from oleomargarine becomes equivalent to the possibility of distinguishing alcohol of 53.7 per cent. from alcohol of 59.2 per cent. As this difference is 5.5 degrees of Gay Lussac's alcohometer, it is very evident that the specific gravity is a sufficient character for distinguishing butter from oleomargarine. This difference may appear more clearly to persons not familiar with alcohometry, by stating that it is the difference between 0.926 specific gravity and 0.915.

By means of the tables of Gay Lussac and of Tralles,\* it is a very easy matter to prepare alcohol of the required strength at any temperature, to be kept in bottles for future use.

As the expansion of fats is different from that of alcohol, it is advisable to bring the alcohol to  $15^{\circ}$  C. when making an observation, which can be easily done by any one provided with a thermometer.

To deliver the sample of fat on the alcohol, I have found that the best plan is to melt the fat and let a large drop of it fall into the liquid. The fat should be melted in a little spoon or a little scoop, and the drop should be delivered by bringing the spoon or scoop close to the surface of the alcohol. It requires a little practice to do this neatly, so

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\* See the excellent tables of Prof. McCulloh.

as not to get an air bubble in the ball of melted fat. When an air bubble becomes imprisoned in the fat, I have had no difficulty in removing it with a strip of paper, while it lies on top of the alcohol. Sometimes the globule of fat only partially sinks in the alcohol; the top of it becomes flat and remains exposed above the liquid. A slight tap on the side of the glass is then generally sufficient to form a wave and sink the globule.

If we take alcohol of  $56\frac{1}{2}$  per cent., which represents equal volumes of alcohol of 53.7 per cent. and of 59.2 per cent., and if we deliver on the surface of this alcohol a globule of melted butter and one of oleomargarine, the butter will sink to the bottom and the oleomargarine will remain at the top, while the two globules are still warm and liquid. Afterwards, if the alcohol has a temperature of about  $30^{\circ}$  C., the butter will become solid, while the oleomargarine may still remain liquid. Then the butter will rise to the top of the alcohol, which is due to the expansion of butter on solidifying. If the alcohol be then kept for a few minutes at  $15^{\circ}$  C., the oleomargarine will become opaque and remain at the top, while the solid globule of butter will sink to the bottom.

If instead of taking alcohol of 56 per cent., we use alcohol of 59.2 per cent., oleomargarine will remain on top, and butter will sink to the bottom at all temperatures above  $15^{\circ}$  C. At  $15^{\circ}$  C., oleomargarine will remain in equilibrium in any portion of the liquid in which it may be placed.

If oleomargarine was always sold pure; the foregoing indications would be sufficient to distinguish it from butter, but the oleomargarine found in the market is always more or less mixed with true butter, to improve its taste and appearance. This being the case, alcohol of 59 per cent. is not the proper liquid to detect oleomargarine. We should use alcohol of 55 per cent., and consider as oleomargarine any so-called butter which will not sink to the bottom in alcohol of this strength at  $15^{\circ}$  C. This is founded on the fact that not more than one-third of butter is ever mixed with oleomargarine to improve its taste and appearance.

Bearing in mind the experiments of Messrs. Leune and Harburet, already cited, the proportion of butter and of oleomargarine in a mixture could be easily detected by finding what strength of alcohol will hold in equilibrium at  $15^{\circ}$  C., a globule of fat under examination. As the difference of 59.2 and 53.7 is 5.5, the proportion of oleomargarine is the difference between the strength of the alcohol and 53.7, divided by 5.5, or more conveniently multiplied by 0.18. If the

alcohol required to hold a globule of fat in equilibrium at 15° C., has a strength of 57 per cent., then:  $(57 - 53.7) \times 0.18 = 3.3 \times 0.18 = 5.95$ , or say  $\frac{1}{10}$  of oleomargarine. If the alcohol had a strength of 58, then  $(58 - 53.7) \times 0.18 = 4.3 \times 0.18 = 7.72$ , or about  $\frac{1}{8}$  of oleomargarine.

The proportions of butter and oleomargarine in a mixture may be also determined without the aid of an alcohometer, by using the two solutions of 53.7 per cent. and of 59.2 per cent. These may be placed in graduated glasses and poured cautiously into a third glass, until an alcohol of sufficient strength is obtained to keep in equilibrium a globule of the fat under examination, at 15° C.

The relative volumes of the two solutions used in making the mixture, give the proportions of butter and oleomargarine.

## XXI.—DETECTION OF STARCH SUGAR SYRUP MIXED WITH SUGAR-HOUSE MOLASSES.

BY P. CASAMAJOR.

In previous communications I have given processes for detecting the adulteration of cane sugar by starch sugar. The adulteration of sugar-house syrups by starch glucose is still more extensively practised than that of sugar, and a great portion of the syrups sold by retailers in this market is mixed with starch glucose.

This form of adulteration may be very easily detected by the use of strong methylic alcohol, in which the alcohometer of Tralles, or of Gay Lussac, will indicate about 93½°.

A straight sugar-house syrup, when mixed with three times its volume of this strong methylic alcohol, will dissolve by stirring, giving a very slight turbidity, which remains suspended, while syrups containing the usual admixture of starch sugar give a very turbid liquid, which separates, when left at rest, into two layers, the lower being a thick viscons deposit containing the glucose syrup.

Considerable quantities are sold of a thin syrup, of about 32° Beaumé, in which the proportion of sugar to the impurities is greater than in common sugar-house molasses. When a syrup of this kind is stirred with three times its volume of methylic alcohol, a marked turbidity and deposition will take place, which consists of pure sugar. The crystals are hard and gritty; they adhere to the sides of the glass, and are deposited on the bottom. There is no resemblance between this precipitate and that due to starch sugar syrup.

It may not be useless to mention, that if a straight sugar-house

syrup, of about 40° Beaumé density, is stirred with three times its volume of ethylic alcohol of about 93½°, the syrup will not dissolve. Hence, ethylic alcohol of this strength is not suitable for distinguishing a syrup mixed with starch glucose syrup from a straight sugar-house syrup.

The presence of starch sugar in sugar-house molasses may be easily detected by the optical saccharometer, when the syrup has the usual density of about 40° Beaumé, and when starch syrup has been added in the usual quantities.

For making the test, the usual weight should be taken (16.35 grms. for Duboscq's saccharometer, and 26.048 grms. for Ventzke's instrument). The direct test should show a percentage of sugar not higher than the number of Beaumé degrees indicating the density, and it may be from 1 to 3 per cent. lower. To understand this we must refer to the composition of cane sugar molasses of 40° Beaumé:

Sugar.....	37.5
Soluble impurities.....	37.5
Water.....	25.0

If the direct test should indicate 55 per cent. of sugar, and if the molasses was straight, the composition would be :

Sugar.....	55
Soluble impurities.....	20
Water.....	25

Now, a product of this composition would not be a clear syrup at 40° Beaumé, but a mixture of syrup and crystals. Therefore, if the product is a clear syrup of 40° Beaumé, and it tests 55 per cent., it cannot be *straight*.—*Q.E.D.*

The presence of starch sugar in a sugar-house molasses may also be detected by the copper test. The possibility of applying this test, as well as those already indicated, rests on the fact that starch glucose is always added in very large quantities for the purpose of adulteration. A small addition could not be satisfactorily detected.

The detection by the copper test rests on the observation that about one-half of the soluble impurities in sugar-house molasses consists of glucose in the shape of inverted sugar. We have seen above that for a molasses of 40° Beaumé, the soluble impurities amount to about 37½ per cent. We may then lay down the rule that the percentage of glucose shown by the copper test cannot, in a straight sugar-house molasses, be much greater than one-half the number expressing the density in Beaumé degrees. The reason is obvious from what has been said of the test by the optical saccharometer.

## XXII.—TABLE FOR THE CORRECTION OF SACCHARIMETRIC TESTS BY INVERSION.

BY P. CASAMAJOR.

In a paper "On the Influence of the Variations of Temperature, on the Deviation of Polarized Light by Solutions of Inverted Sugar," published in 1879,\* I endeavored to show the principle on which is founded the method, so important in sugar analysis, of correcting the direct test of the optical saccharometer, by taking into account the test after inversion. In this communication I will assume that the reader is familiar with the subject, and I propose to enter only very briefly into it.

Clerget, to whom this method of correction is due, published a table in the *Annales de Chimie et de Physique* (26, 3d series, 175), by means of which the desired rectification may be obtained, without the need of any calculation beyond taking the algebraical difference of the indications of the saccharometer, before and after inversion. There is also a table by Dr. Tuchschnid, which is essentially the same as Clerget's. Although these tables are published in several works on sugar, many persons who need to use them find great difficulty in obtaining them. There is the resource of copying them by hand, but they are so extensive that this would involve an amount of labor which few persons like to undertake.

The following table will be found useful by persons who have not those spoken of. It is very short and it would be little trouble to copy it. It requires but one operation more than Clerget's table, which is to multiply the algebraical difference of the two readings by a certain factor, corresponding to the temperature of the inverted sugar at the time of observation.

To understand how the numbers in this table were obtained, we must bear in mind that the deviation due to inverted sugar varies with the temperature. For a solution of inverted sugar giving a deviation of 100 to the right, the indication, after inversion, is 44 to the left, or —44, if the observation is made at 0° C. For every centigrade degree above 0, the deviation becomes half a degree less of the

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\* See JOURNAL OF THE AMERICAN CHEMICAL SOCIETY, I, 26; also *Chemical News*, 39, 212, 234; *The Sugar Cane*, II, 296; *Moniteur Scientifique*, Juin, 1879, 647; *Zeitschrift des Vereins*, July, 1879, 683.

90 TABLE FOR CORRECTION OF SACCHARIMETRIC TESTS BY INVERSION.

negative scale. If we call the temperature  $t$ , the law of deviations after inversion is expressed by

$$-\left(44 - \frac{t}{2}\right)$$

For a temperature of  $10^{\circ}$  C., the deviation is  $-39$ ; for  $20^{\circ}$  C., the deviation is  $-34$ , etc.

If  $D$  is the indication of the saccharometer before inversion, and  $-D'$  the indication after inversion,  $D + D'$  will be the algebraical difference, and, for a solution of pure sugar, with  $D = 100$ ,

$$D + D' = 144 - \frac{t}{2}$$

If the direct test is not 100, and if we call the correct test  $Z$ , we will have

$$\frac{Z}{D + D'} = \frac{100}{144 - \frac{t}{2}}$$

$$\text{Whence } Z = D + D' \times \frac{100}{144 - \frac{t}{2}}$$

If for every degree centigrade, we calculate this quantity :

$$\frac{100}{144 - \frac{t}{2}}$$

we may form a table by writing it opposite to every degree of temperature. We will then have in a second column the factors by which to multiply the difference of the indications, before and after inversion, to obtain correct results.

**NUMERICAL EXAMPLE.**—If the direct reading of the saccharometer is 90, and, after inversion,  $-27$ , the temperature being  $25^{\circ}$  C., then  $D + D' = 117$ . Referring to the table, we find, opposite  $25^{\circ}$  C., the factor 760. This number multiplied by 117 gives 88.9, which is the correct result.

This table is given on next page :

TABLE OF FACTORS CORRESPONDING TO  $\frac{100}{144 - \frac{t}{2}}$

<i>Degrees Centigrade.</i>	<i>Factors.</i>	<i>Degrees Centigrade.</i>	<i>Factors.</i>
10°.....	0.719	26°.....	0.763
11.....	0.722	27.....	0.766
12.....	0.724	28.....	0.768
13.....	0.727	29.....	0.771
14.....	0.730	30.....	0.774
15.....	0.732	31.....	0.777
16.....	0.735	32.....	0.780
17.....	0.738	33.....	0.784
18.....	0.740	34.....	0.787
19.....	0.743	35.....	0.790
20.....	0.746	36.....	0.793
21.....	0.749	37.....	0.796
22.....	0.752	38.....	0.800
23.....	0.754	39.....	0.803
24.....	0.757	40.....	0.806
25.....	0.760		

XXIII.—AN APPARATUS FOR THE RAPID ANALYSIS OF MIXTURES OF GASES.

BY ARTHUR H. ELLIOTT, PH.B, F.C.S.

The apparatus is of the type of those in which the various gases are absorbed by the use of liquid reagents added in a certain order. This was carried out by Raoult\* in a graduated tube with a stop-cock above and another below, and the upper stop-cock surrounded by a cylindrical funnel. But the washing out of each reagent before adding a new one, and a somewhat risky manipulation of the tube and stop-cocks to insure measuring the gas without pressure, makes this method troublesome. Wilkinson further modified this method of Raoult, by introducing another vessel in which the burette stood in water, doing away with the lower stop-cock. By this means, the level of the water in the burette, and the exterior tube, could be



without pressure. This was an improvement over the method of Raoult, but it still left the washing out of the reagents as a drawback.

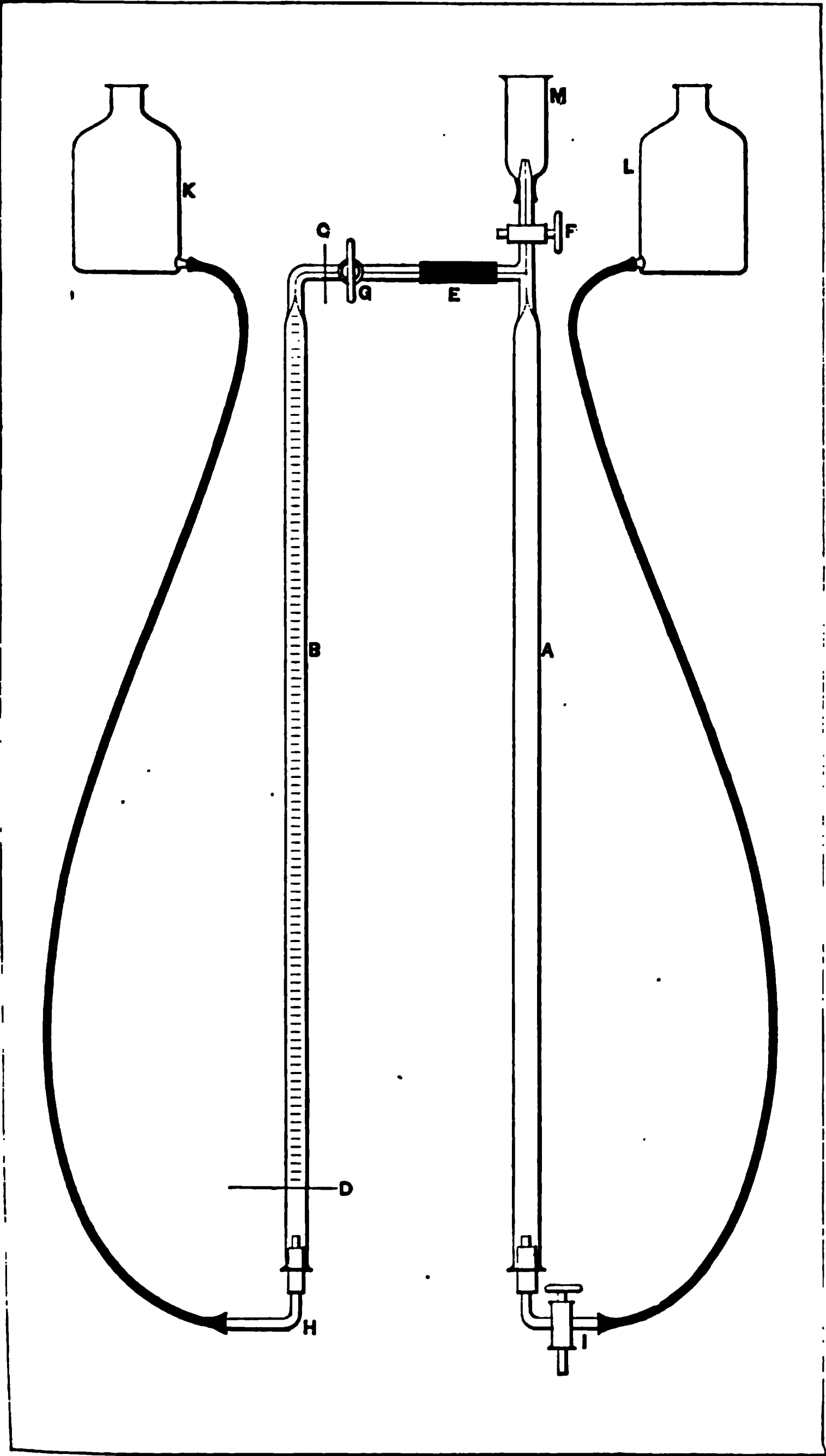
This washing becomes very important in many cases. For example, if we have a mixture of gases rich in olefient gas and ethylene, and after treating with potassic hydrate and pyrogallate of potassium, we wash out the alkali, before adding bromine to absorb the illuminants (olefient gases). The water necessary to remove this alkali, will reduce the volume of the illuminants as much as two per cent.

In the apparatus I have devised, the gas is removed from the absorbant liquid and measured in another vessel, without washing.

The apparatus is shown in the accompanying drawing. The tube A is of about 125 c.c. capacity, whilst B, although the same length, holds only 100 c.c. from the point D, or zero, to the mark on the capillary tube at C, and is carefully graduated in  $\frac{1}{16}$  c.c. The attachments to these tubes below are seen from the drawing, except that the stop-cock I is three-way and has a delivery through its stern. The bottles K and L hold about 1 pint each. The tubes A and B are connected above with one another, and also with the cylindrical funnel M, by a series of capillary tubes about 1 millimeter in diameter inside. There is a stop-cock at G and another at F, whilst the funnel M, which holds about 60 c.c., is ground to fit over the end of F above. At F is a piece of rubber tubing uniting the ends of the capillary tubes, which are filed square to make them fit as close as possible.

In beginning the analysis of a mixture of gases, the stem exit of the three-way cock I is closed by turning it so that L and A are connected through the rubber tubing; the stop-cocks F and G are opened, and water is allowed to fill the apparatus from the bottles K and L, which have been previously supplied.

When the water rises in the funnel M, and all air-bubbles have been driven out of the tubes, the stop-cocks F and G are closed, the funnel M removed, and the tube delivering the gas attached in its place. By now lowering the bottle L slowly, and simultaneously opening the stop-cock F, the tube A is nearly filled with gas, and the stop-cock F is closed. The tube delivering the gas is removed, the funnel M replaced, the bottle L raised, the bottle K lowered, and by opening the stop-cock G, the gas is transferred to the graduated tube B. By placing the bottle L on a stand at about the height of the water in A, the level in B and in the bottle K can be adjusted to the



zero point, and the stop-cock G is closed. The excess of gas in A is expelled by opening the stop-cock F and raising the bottle L. The gas remaining in the capillary tube between C and the vertical part is disregarded, or its value may be ascertained and an allowance made; but usually it is too trifling to be worth notice.

Having measured the gas, it is now transferred by means of the bottles K and L into the tube A, and the fluid chemicals added by placing them in the funnel M and allowing them to flow down the sides of the tube A slowly, care being taken *never* to let the fluids run below the level of the top of the vertical tube in the funnel. It is best to have a mark on the outside of the funnel at least three-fourths of an inch above the top of the level of the vertical tube, and never draw the fluid down below this point.

Having treated the gas with the chemical, it is transferred by means of the bottles, to the tube B to be measured. If the chemical gets into the horizontal capillary tube, the passage of a little water from the bottle K will remove it, before transferring the gas. When the gas residue is in B, and the fluid of A has been adjusted at the mark C on the horizontal tube, the stop-cock G is closed, the bottle K is lowered till the level of liquid in it and in the tube B are the same, and the reading is then made. The tube A is now filled with the chemical, just used as absorbant, and water, by turning the stem of the three-way cock I, so that it communicates with A, and also opening the stop-cock F, the contents of the tube can be run out, and water run through the funnel M to clean the tube for a new absorption. When the tube is clean, by turning the stop-cock I so that A and L are connected, the water is poured into A, and the whole is ready to receive the gas in B for new treatment.

By this method a mixture containing carbonic acid, oxygen, illuminants and carbonic oxide, can be analyzed in from 20 to 30 minutes, according to the amount of practice the operator has had with the apparatus.

Compared with Orsat's apparatus, the work can be done with the above described apparatus in one-fourth the amount of time, and with identical results.

The water used in the apparatus should have the same temperature as the room in which the analysis is made; and by careful handling, little or none of the chemicals get into the bottle L. When working in a warm place, the tube B should be surrounded with a water jacket to prevent change of volume in the gas while under treatment.

School of Mines, Columbia College, N. Y.

# THE AMERICAN CHEMICAL SOCIETY.

## XXIV.—PROCEEDINGS.

*Regular Meeting, October 7th, 1881.*

The meeting was called to order at 8:20 P. M., Prof. A. R. LEEDS in the chair.

The minutes of the previous meeting were read and approved.

The election of new members was then deferred till after the reading of the first paper, in order to prepare the ballots.

In the meanwhile the following gentlemen were nominated as new members :

Dr. C. W. VOLNEY.

Proposed by { WM. RUPP,  
M. BENJAMIN,  
JAMES H. STEBBINS, JR.

Prof. R. A. WITTHAUS.

Proposed by { CHAS. A. DOREMUS,  
JAS. H. STEBBINS, JR.,  
WM. RUPP.

C. E. MUNSELL.

Proposed by { ARTHUR H. ELLIOTT,  
JAMES H. STEBBINS, JR.,  
T. O'C. SLOANE.

NELSON H. DARTON.

Proposed by { JAMES H. STEBBINS, JR.,  
T. O'C. SLOANE,  
ARNO BEHR.

W. W. SHARE.

Proposed by { S. A. GOLDSCHMIDT,  
ARTHUR H. ELLIOTT,  
JAMES H. STEBBINS, JR.

The first paper of the evening, "On the Comparative Purity of City Water," by Dr. A. R. LEEDS, was then read.

Some questions by Messrs. ENDEMANN, BEHR, CASAMAJOR and SQUIBB then followed.

As the ballots were now ready, the following gentlemen were voted for, and duly elected as active members :

ALBERT L. COLBY.

Proposed by { ARTHUR H. ELLIOTT,  
ELWYN WALLER,  
A. R. LEEDS.

Dr. WINFIELD P. LAUVER.

Proposed by { BENJ. S. HEDRICK,  
P. CASAMAJOR,  
H. ENDEMANN.

Dr. HARRY VON BAUER.

Proposed by { H. ENDEMANN,  
JAMES H. STEBBINS, JR.,  
P. CASAMAJOR.

Dr. P. RADENHAUSEN.

Proposed by { H. ENDEMANN,  
JAMES H. STEBBINS, JR.,  
P. CASAMAJOR.

Dr. LOUIS HABEL.

Proposed by { H. ENDEMANN,  
J. P. BATTERSHALL,  
M. BENJAMIN.

H. C. HIEPE.

Proposed by { H. ENDEMANN,  
JAMES H. STEBBINS, JR.,  
J. F. BATTERSHALL.

WM. L. LEMAN.

Proposed by { T. J. PARKER,  
P. CASAMAJOR,  
E. WALLER.

After which the second paper of the evening, "Upon some New Salts of Thymole Sulpho-acid, and some New Facts concerning the same," by JAMES H. STEBBINS, JR., S.B., was then read.

After some questions by Dr. LEEDS, the third paper of the evening, "On the Description of some New Gas Apparatus," by Dr. W. HEMPEL, then followed.

In the course of his description, samples of gas and air were analyzed, thus showing the efficiency of his instruments.

Mr. CASAMAJOR then proposed that a committee, to nominate officers for the coming year, be elected.

It was finally moved, that each person should write out the names of three candidates to serve as Nominating Committee.

This, on motion, was unanimously carried.

The following officers were then elected to serve on this committee: H. ENDEMANN, WM. GEYER, and A. R. LEEDS.

After which the meeting adjourned.

JAMES H. STEBBINS, JR.,  
*Recording Secretary.*

## MEETING OF THE BOARD OF DIRECTORS.

*Held Sept. 28th, at Wm. H. Nichols' Office, 41 Cedar St., N. Y.*

The following Directors were present: A. R. LEEDS, M. ALSBERG, E. R. SQUIBB, W. H. NICHOLS, H. ENDEMANN, JAMES H. STEBBINS, JR.; Dr. A. R. LEEDS in the chair.

The minutes of previous meeting were read and approved.

The Recording Secretary had no report.

The Treasurer then announced, that we have a balance on hand of about \$400, from which we have to draw: \$155.69 for printer; \$25 for janitor; \$60.50 for rent.

The Treasurer then moved, that the requisite amount be appropriated, with which to meet these bills.

On motion, this was unanimously carried.

The question of printing a new list of members was then brought up, and ended in the following motion by Dr. LEEDS:

That if the funds permit, a list of members, combined with an index of patents, be published about the 15th of Nov., 1881, along with the JOURNAL.

This being moved and seconded, was carried unanimously.

The Committee on Papers and Publications then stated that only 500 copies of the journal were printed the last time, leaving over about 100 extra copies. This making a difference of about \$25 in the Society's favor.

Dr. LEEDS then announced the following paper for the next meeting: "On the Comparative Purity of City Water," by A. R. LEEDS, Ph.D.

Dr. ENDEMANN then stated, that possibly Dr. HEMPEL might favor us with an exhibition of his gas apparatus.

The Committee on the Endowment Fund then stated, that a subscription of \$50 extra had been made, and that total subscriptions, up to date, amount to about \$8,000 or \$9,000.

*Deferred business:* Some comment about the coming elections took place here. Mr. NICHOLS then moved, that this year's elections be carried on in the usual way. On motion, this was unanimously carried.

After which the meeting adjourned.

JAMES H. STEBBINS, JR.,  
*Recording Secretary.*

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**XXV.—RELATIVE PURITY OF THE CITY WATERS IN THE  
UNITED STATES.**

BY DR. A. R. LEEDS.

A legal suit has recently been instituted, which will probably have a very important bearing in the future upon the quality of water supplied to many of the principal cities in the United States. To understand more readily the nature of the questions raised in this suit, a few words of previous explanation will be requisite. The three cities of Newark, Jersey City and Hoboken, with an aggregate population of 300,000 inhabitants, all draw their water supply from the river Passaic. This is a large stream, having a daily flow at Newark of about 100,000,000 gallons. Its water-shed is mainly granitic in character, and occupies about 950 square miles in the northern part of the State of New Jersey. About three miles above the city of Newark, the Belleville pumping stations are located, and at this point the supplies of the three above-mentioned cities are taken. Some eighteen miles farther up the stream is the town of Paterson, a community almost entirely engaged in the manufacture of paper, cotton, woolen and silk goods, locomotives, etc., and which throws its entire refuse and the sewage of 50,000 inhabitants into the Passaic. Above Paterson there are few villages and factories, so that the water going over the great falls at this place is of superior quality for drinking purposes. Below Paterson manufactories are gradually springing up, and are a constant source of uneasiness and alarm to the large population using the water as pumped at Belleville. During the course of the year just past, one of these factories, an extensive paper mill, situated on the Third River, a small tributary emptying into the Passaic a short distance above Belleville, threw into the water the refuse derived from washing out some carbolized paper. The taste and odor of carbolic acid were so strong that they caused a temporary suspension of the use of the Newark, Jersey City and Hoboken water for drinking. The Newark Aqueduct Board summoned the parties to desist, which they did at the time, but afterwards repeated the offence, when suit was brought against them by the Board. In defence, the plea was entered, and an attempt made to substantiate it by the testimony of many manufacturers, that the water was already so badly polluted at Paterson that it could not be made more unfit for drinking purposes by the introduction of some carbolic acid. In fact, it was urged that, so far from being rendered

any worse from the carbolic acid, it was actually made better, for the reason that the carbolic acid served to purify and disinfect it. The case was decided in favor of the Aqueduct Board. The importance of having successfully established such a precedent can scarcely be overestimated in connection with its future influence upon the sanitary welfare of rapidly growing communities. In this category are to be placed the large cities in the Eastern and Middle States, nearly all of which are contending with one phase or another of the evils arising from the crowding in of population, and a population in many instances devoted to manufacturing, upon their source of water supply. As a consequence, the problem whether to abandon the original sources, which in most cases are those designed by nature, and to seek more distant and expensive ones, or to restrain the population by legal processes, is most urgent.

In this connection, the author has been asked by the Boards of Commissioners of Newark and Jersey City to examine into the amounts of impurities in the river Passaic, beginning with a point above Paterson, and making analyses of samples collected at an interval of a mile, until the stream has passed the city of Newark. The collection was made all upon the one day, at the close of a period of many weeks of almost unexampled drought. The analysis of the forty samples thus collected will show not only the composition of the river water, in its worst condition, but likewise the changes in that composition during a flow of twenty-one miles.

Connected with this inquiry was the important one of determining the quality of the water supply, as compared with that of other cities. The design was to collect the samples on the same day, and analyze them all by the same methods. It was but partially successful, owing to a failure to obtain simultaneous co-operation in so many places. In the case of Washington, this was due to the fact that Dr. Smith Townshend, the health officer of the district of Columbia, at the very time of receiving my telegraphic request, was summoned to attend the wounded President in the railroad station at Washington. In the great excitement and general distress, my telegram was naturally lost sight of for several days. Neither was it a mode of comparison unexceptionable in point of principle, inasmuch as the climatic conditions were not exactly the same in the various places. So that from the results gathered, it would be manifestly unfair to state that the order of purity was fixedly maintained as that given. It could only be said that at the time when the comparison was made, about the 1st of July of this year (1881), such an order existed.



[illegible]

Together with these determinations, a number are given of the water of the river Passaic as delivered at widely different points, and at nearly the same hour, on the same day, in Jersey City and Hoboken. They show not inconsiderable differences in composition, and illustrate to what extent water may differ when flowing at very different rates of velocity, or in the case of dead ends scarcely at all, in one and the same system of distributing pipes. Nearly all these pipes were of iron, cement pipes being little used. The results given are in parts per 100,000.

The labor of making a comparison was greatly facilitated by plotting these results on a large chart, so as to form a graphic representation of them.\* From this, the following order of relative purity was inferred:

- I. Brooklyn.
- II. Rochester.
- III. Philadelphia.
- IV. Baltimore.
- V. Washington.
- VI. New York.
- VII. Newark, Jersey City, Hoboken.
- VIII. Cincinnati.
- IX. Oswego.
- X. Wilmington, Delaware.
- XI. Boston.

In many particulars the order thus arrived at was quite unforeseen, and was a matter of great surprise. This was more especially true of Boston, which I anticipated would stand nearly at the head of the list, but which actually came at the bottom. And yet the water-sheds of both Brooklyn and Boston are on drift gravel. A month later I found that the best hotels in Boston, and private citizens who were willing to bear the extra expense, were supplied by spring water carted into the city, and sold at the rate of 10 cents per gallon. This state of affairs continued until the 28th of August, when I visited the city again, and inspected the Cochituate Lake and the Sudbury and Mystic Rivers, the three sources of supply. The water in the reservoirs supplied by the two latter streams had a yellow color and disagreeable, nasty taste and smell. During the earlier portion of the summer, all of the reservoirs had contained an abundant growth of algæ, which later on had decomposed, leaving

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\* The oxygen required to oxidize the organic matters was determined with potassium permanganate at 100° C.

behind the bad taste referred to. A striking peculiarity of these Boston waters is the large amount of decomposable organic matters held in solution. These points will best appear by examination of the results of the following analyses of samples collected on the 29th and 30th of August:

	<i>Mystic Lake.</i>	<i>Sudbury River. Farm Pond.</i>	<i>First Dam.</i>	<i>Cochituate.</i>	<i>Pegan Brook.</i>
Free ammonia.....	0.015	0.001	0.012	0.025	0.289
Albuminoid ammonia.....	0.032	0.0325	0.039	0.064	0.240
Oxygen required to oxidize					
organic matters.....	0.900	1.180	1.190	0.830	6.630
Chlorine.....	3.010	0.460	0.440	0.460	7.650
Total solids.....	12.500	7.500	7.500	7.500	49.000
Mineral water.....	8.000	4.000	4.000	5.000	24.000
Organic and volatile matter	4.500	3.500	3.500	2.500	25.000

The causes of the inferior character of the Boston water supply are, first, its pollution by a few manufacturing towns, and, secondly, the growth of algæ, which again is favored by the shallow character of portions of the reservoirs and impounded streams. The worst of these sources of pollution is Pegan Brook, which flows through the town of Natick, a town of ten thousand inhabitants, almost entirely engaged in the manufacture of shoes, and empties into Cochituate Lake. The attempt has been made to mitigate the evil by throwing two filtering dams across the outlet of the stream, but both the forebays thus formed are densely overgrown with the lowest forms of aquatic vegetation, and may, to a certain extent, act as nurseries for the growth of these plants.

The Rochester water supply is derived from Hemlock Lake, remote from all sources of contamination, and after flowing through conduits for a number of miles, is discharged through a noble fountain into a reservoir near the city. A large portion of the Philadelphia supply is obtained from the river Schuylkill. At one time it was in imminent danger of fatal pollution from the growth of manufactories on the borders of the stream. But the city purchased the lands on both banks, nearly to the manufacturing town of Manyunk, located about seven miles above, and included these lands in a great park, so as to keep back all buildings from the water's edge. New York and all the places mentioned lower on the list receive their water supply from contaminated sources. The feeders which empty into Croton Lake, the principal reservoir of the New York water, pass through a settled country, with numerous tanneries, factories,

etc., along their banks. Analyses of the Croton water, made at different seasons during the past five years, have shown that it is to be classed among contaminated water supplies.

On the publication of the results above stated, it was indignantly claimed by the newspaper press of Wilmington, Delaware, that an injustice had been done to their city. But subsequently, letters published in reply by citizens of the place, have affirmed the correctness of the judgment founded on the analytical data, and have enumerated the various breweries, woolen, cotton and other mills and dye-houses, as affording a sufficient explanation of the low position assigned to the water supply.

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## XXVI.—SOME NEW FACTS CONCERNING THYMOLE SULPHO-ACID, AND SOME OF ITS SALTS.

BY JAMES H. STEBBINS, JR.

This work was undertaken for two reasons.

*First*, to establish certain facts, which thus far have either been neglected or overlooked; as for example, the production of free thymole sulpho-acid, which hitherto has only been obtained in combination with other salts. It was also my intention to produce from this latter, by direct combination, the calcium, ammonium, and soda salts, all of which have thus far not been mentioned. Or starting from, say, the barium salt, to produce by indirect combination either of the previously mentioned salts.

*Second*, to combine these new bodies with diazo compounds, and thus ascertain whether it is possible to produce azo compounds, from phenoles of this description.

This hypothesis seems all the more probable, as only a short time since, I obtained a yellow dye-stuff, by the direct combination of diazobenzole nitrate with an alkaline solution of thymole. As this dye was insoluble in water, the usual method of rendering such compounds soluble was resorted to, namely, the introduction of the sulpho group into the benzole nucleus.

My experiments in this line will be detailed in a subsequent paper.

The first person to engage in the study of these compounds was Lallemand,\* who obtained a thymole sulpho-acid, by treating thymole with hydrochlor-sulphuric acid ( $\text{SO}_3\text{HCl}$ ).

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\* *Jahresber. f.* 1856, 617.

In 1869, A. Engelhardt and P. Latschinoff\* produced three thymole sulpho-acids, by the action of sulphuric acid upon thymole, at a temperature of about 50° C. These three sulpho acids were only obtained in combination with barium, potassium, copper, lead, etc., thus making distinct, and well characterized salts, but making no attempt to produce the free sulpho acids.

It seemed, therefore, advisable to investigate this subject, and in so doing the following experiments were made :

60 grms. of thymole were treated in an evaporating dish with 50 grms. of sulphuric acid.† The dish was placed upon a steam bath, and heated for about 15 minutes.‡

The thymole soon began to dissolve, and was constantly stirred, till suddenly nearly the whole contents of the dish solidified out into a pink, crystalline mass. This mass was then drained from a clear and colorless oily fluid which impregnated the whole crystalline body.

This oily liquid solidified into a white, crystalline mass, as soon as it came in contact with the cold sides of the glass. A few isolated crystals were examined with a magnifying glass, and showed themselves to be composed of star-shaped needles. They were then labeled and set aside for further examination.

The pink crystalline mass was then dissolved in boiling water, neutralized with carbonate of calcium, and filtered hot. The sulphate of lime thus formed, besides a small quantity of unattacked thymole, were thus gotten rid of. The filtrate so obtained is nearly colorless, and was concentrated over the steam bath, till it had assumed the consistency of a syrup. On cooling, the calcium salt of alpha thymole sulpho-acid crystallized out in rhombic plates.

The solid crystalline mass so obtained was separated from the mother liquor, and dissolved at a gentle heat in 90 per cent. alcohol. The larger part of the alcohol was distilled off, and the syrup thus formed allowed to crystallize. The salt so obtained crystallizes in long white, flexible needles, bearing a strong resemblance to asbestos filaments. If a drop of the above alcoholic solution be rapidly evaporated on a watch-glass, a large number of minute star-grouped needles are obtained. These viewed under the polariscope, show a beautiful effect of circular polarized light. The phenomenon

\* *Zeitschrift f. Chem.*, 1869, 43; and 615.

† The acid used was the common 66° acid.

‡ In this instance I used a higher temperature than Engelhardt and Latschinoff.

observed is a symmetrical black cross, when the nicols are crossed, with a series of concentric rings, dark, and light in monochromatic light; but, in white light, showing the prismatic colors in succession in each ring.

The cross becomes white when the nicols are in a parallel position, and each band of color in the white light changes to its complementary tint, thus making the uniaxial character of the crystal.\*

The crystals so obtained were then redissolved in water, and allowed to crystallize. This salt crystallizes in two forms of rhombic plates.

The first, showing the planes  $mc: \infty b: a$ ; and  $mc: b: \infty a$ ; or macro and brachydomes.

The second, showing the planes  $mc: b: \infty a$ , or brachydomes, and  $\infty a: b: \infty c$ ; or macropinacoids.

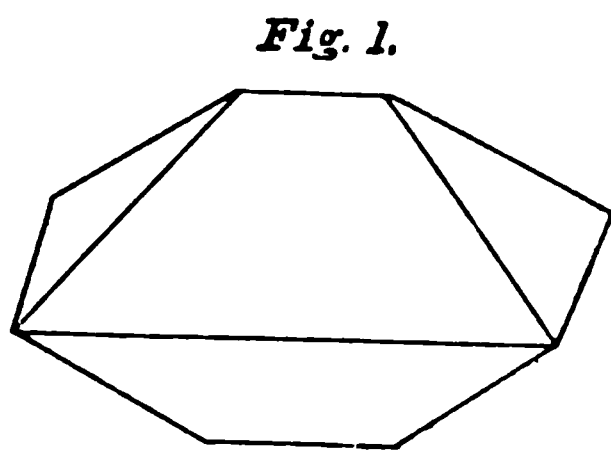


Fig. 1.

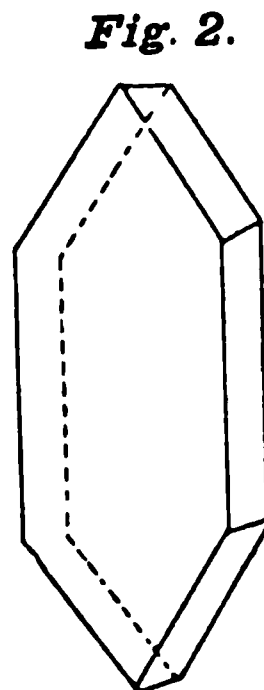


Fig. 2.

Both these crystals are the same identical compound, and constitute the calcium salt of alpha thymole sulpho-acid.

A sulphur estimation shows the correctness of this statement.

0.4246 grm. of substance yielded 0.3948 grm.  $\text{BaSO}_4$ , which is equivalent to :

Sulphur..... 12.77 per cent.

#### THEORY.

Sulphur..... 12.85 per cent.

Likewise a lime estimation gave:

Calcium ..... 9.34 per cent.

\* These phenomena seem to be totally ignored by Engelhardt and Latschinoff, and I therefore look upon them as new, and worthy of a position in the literature of thymole and its substitution compounds.

## THEORY.

Calcium ..... 8.32 per cent.

These figures lead us, beyond doubt, to the formula:



The above compound dried at 100° C., loses two molecules of water, and falls into a fine white powder. Melting point, 156 to 157° C.

It is easily soluble in water, alcohol and ether.

## MOTHER LIQUOR.

The mother liquor from the first crop of crystals was poured upon a watch-glass, and after a short time crystallized in rhombic plates, showing respectively the same form, melting point and sulphur percentage, as in the previous compound. It was, therefore, considered identical

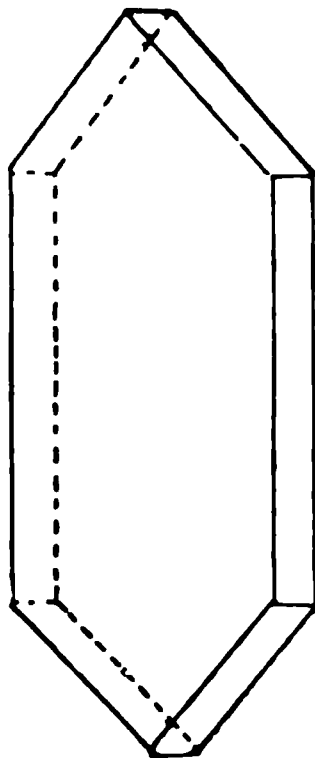
## AMMONIA SALT.

A part of the Ca salt of alpha thymole sulpho-acid was next dissolved in water, heated to boiling, and decomposed by  $(NH_4)_2CO_3$ . The  $CaCO_3$  formed was then filtered off, and the filtrate concentrated over the steam bath, to the consistency of a syrup.

The syrup so produced was then placed under the air pump, and allowed to crystallize.

After standing for a few hours, the fluid became filled with pretty, crystalline white plates. These were then drained from the mother liquor, and dried between filter paper.

The salt so obtained crystallizes in white rhombic plates, with strong vitreous lustre, and shows the planes  $mc: \infty a: b$ ; or brachydomes; and  $\infty a: b: \infty c$ ; or macropinacoids.



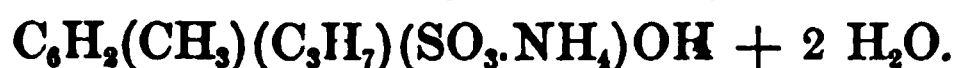
At the ordinary room temperature it loses a part of its water of crystallization, and at 100° C. all the water is driven off, while the substance crumbles into a fine white powder.

1.9748 gram. of substance, on being heated to 100° C., lost 0.3794 gram. of water, which is nearly equivalent to 2 molecules.

Therefore, the ammonium salt of thymole sulpho-acid crystallizes with 2 molecules water of crystallization.

Melting point, 172 to 172.5° C.

Therefore, this salt may be represented by the following formula:



The soda salt, as well as an attempt at producing the free alpha thymole sulpho-acid, will be described in a subsequent paper.



# THE AMERICAN CHEMICAL SOCIETY.

## XXVII.—PROCEEDINGS.

*Regular Meeting, Friday, November 4th, 1881.*

The meeting was called to order at 8:40 P. M., Prof. A. R. LEEDS in the chair.

The minutes of the previous meeting were read and approved.

After which the following gentlemen were elected as active members:

Dr. C. W. VOLNEY.

Proposed by { WM. RUPP,  
M. BENJAMIN,  
JAMES H. STEBBINS, JR.

Prof. R. A. WITTHAUS.

Proposed by { CHAS. A. DOREMUS,  
JAMES H. STEBBINS, JR.,  
WM. RUPP.

C. E. MUNSELL.

Proposed by { A. H. ELLIOTT,  
JAMES H. STEBBINS, JR.,  
T. O'C. SLOANE.

NELSON H. DARTON.

Proposed by { JAMES H. STEBBINS, JR.,  
T. O'C. SLOANE,  
ARNO BEHR.

W. W. SHARE.

Proposed by { S. A. GOLDSCHMIDT,  
A. H. ELLIOTT,  
JAMES H. STEBBINS, JR.

THOS. D. O'CONNOR.

Proposed by { T. O'C. SLOANE,  
P. CASAMAJOR,  
JAMES H. STEBBINS, JR.

As there were no new members to nominate, the next in order was the report from the Nominating Committee.

This had to be deferred, however, as the chairman of said committee was absent.

The Recording Secretary then announced the resignation of Dr. WILEY, which, on motion, was duly accepted.

The Secretary then announced the death of Mr. CHAS. SCHMICH, who died last March of pneumonia.

It was then moved that Mr. SCHMICH's death be duly entered on the minutes. This being seconded, was carried unanimously.

After which the first paper of the evening, "On some New Salts of Alpha Thymole Sulpho-acid, and some New Facts concerning the same," by JAMES H. STEBBINS, JR., S.B., was then read.

After some questions by Mr. ELLIOTT, the second paper, "On the Combination of Diazo Compounds with Thymole Sulpho-acid," by JAMES H. STEBBINS, JR., S.B., then followed.

After some questions by Messrs. ELLIOTT and FRIEDBURG, the third paper, "On the Constitution of the Explosive Derivatives of Glycerine," by Dr. VOLNEY, was then read.

Some criticisms and remarks by Dr. FRIEDBURG and others then followed.

The report of the Nominating Committee was now ready, and the following gentlemen were nominated as candidates for the coming election :

*Corresponding Secretary :*

P. CASAMAJOR.

*Recording Secretary :*

JAMES H. STEBBINS, JR.

*Librarian :*

GEO. PROCHAZKA.

*Curators :*

ROSSI, FESQUET, RUPP.

*Committee on Papers and Publications :*

ARNO BEHR, A. R. LEDOUX and H. ENDEMANN.

*Committee on Nominations :*

ARTHUR H. ELLIOTT,  
O. H. KRAUSE,

J. P. BATTERSHALL,  
J. B. F. HERRESHOFF,

T. O'C. SLOANE.

*Directors :*

P. CASAMAJOR,	JAMES H. STEBBINS, JR.,	H. MORTON,
C. F. CHANDLER,	M. ALSBERG,	E. R. SQUIBB,
W. H. NICHOLS,	W. M. HABIRSHAW,	ELWYN WALLER,
H. ENDEMANN,	A. R. GALLATIN,	GEO. PROCHAZKA,
	A. R. LEEDS.	

As the Chairman was obliged to depart before the close of meeting, Mr. CASAMAJOR was elected Chairman *pro tem*.

The meeting was then adjourned.

JAMES H. STEBBINS, JR.,  
*Recording Secretary.*

XXVIII.—ON SOME NEW SALTS OF ALPHA THYMOLE SULPHO-ACID,  
AND SOME NEW FACTS CONCERNING THE SAME.

(Second Paper.)

BY JAMES H. STEBBINS, JR.

In my last paper I described the calcium and ammonium salts of alpha thymole sulpho-acid. In this paper I will describe the sodium salt and free sulpho acid, which I mentioned in connection with my first paper.

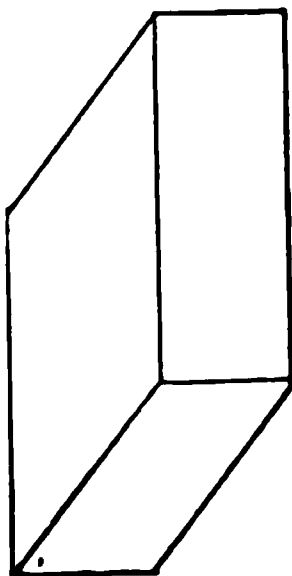
Soda salt of alpha thymole sulpho-acid,



A part of the lime salt was dissolved in boiling water and decomposed with  $\text{Na}_2\text{CO}_3$ .

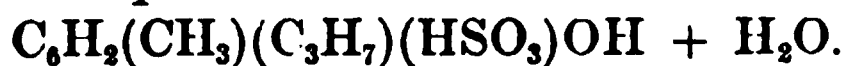
The  $\text{CaCO}_3$  thus formed was then filtered off, and the clear filtrate concentrated over the steam bath to the consistency of a syrup. On cooling, the soda salt crystallized in fine white, rhombic plates, with strong vitreous lustre. These crystals show the planes,  $a : \infty c : \infty b$ , or macropinacoids ; and  $\infty c : b : \infty a$ , or macrodomes.

The crystalline form may be seen from the following diagram:



This compound is easily soluble in water and alcohol; melting point, 166 to 167° C. At 100° C., it parts with all its water of crystallization, and crumbles into a fine white powder, in the same manner as the other salts described.

*Alpha thymole sulpho-acid:*



It was first tried to obtain this body by decomposing the lime salt with  $\text{CO}_2$ ; however, this failed completely, as even a prolonged passing of  $\text{CO}_2$  through a concentrated solution of the lime salt failed to produce the slightest precipitate of carbonate of lime. Therefore, this was abandoned, and the lead salt was resorted to, as the best means of accomplishing this end. 60 grms. of thymole were dissolved in 50 grms. of  $\text{H}_2\text{SO}_4$ , and heated on the steam bath till the contents of the dish became nearly one crystallized mass. The colorless fluid \* remaining was poured off, and the pink crystalline body dissolved in boiling water, and was then neutralized with carbonate of lead. The lead sulphate formed was filtered off, and a current of  $\text{H}_2\text{S}$  was then passed through the clear filtrate, till all the lead (in combination with thymole sulpho-acid) had been precipitated as black sulphide of lead. This was then collected on a filter, and the filtrate evaporated to the consistency of a syrup. When cold, the free sulpho acid crystallized in beautiful, large white rhombic scales, with pearly lustre and greasy feel.

It dissolves very readily in hot and cold water; melting point, 91 to 92° C.

This body, in crystallizing, retains a certain amount of water, with which it parts, however, if placed for a few days over sulphuric acid.

A water estimation of substance dried over sulphuric acid gave: 1.052 grm. substance lost 0.15 grm. of water, or equivalent to very nearly one molecule.

Heated on platinum foil, the salt first melts and then burns with a sooty red flame, leaving a voluminous black carbon residue behind.

A carbon and hydrogen estimation of the above gave:

	THEORY.		
$\text{C}_{10}$ .....	120	.....	52.17 per cent.
$\text{H}_{14}$ .....	14	.....	6.08 “
$\text{S}$ .....	32	.....	13.91 “
$\text{O}_4$ .....	64	.....	27.84 “
	<hr/> 230		<hr/> 100.00

\* This fluid is the same as mentioned in my first, as a by-product in the preparation of alpha thymole sulpho-acid.

## FOUND.

Carbon.....	52.19 per cent.
Hydrogen.....	6.10 “

## BY-PRODUCT.

The by-product heretofore described was dissolved in boiling water, and neutralized with  $\text{PbCO}_3$ .

The precipitate of  $\text{PbSO}_4$  was collected on a filter, and the clear filtrate treated with a current of  $\text{H}_2\text{S}$ , till all the lead had been thrown down as  $\text{PbS}$ . This was collected on a filter, and the filtrate evaporated to the consistency of a syrup. It was then placed over sulphuric acid and allowed to crystallize. The compound so obtained crystallizes in long, white needles, which bear a striking resemblance to the disulpho acid of alpha thymole. They are very easily soluble in water, and are likewise quite hygroscopic.

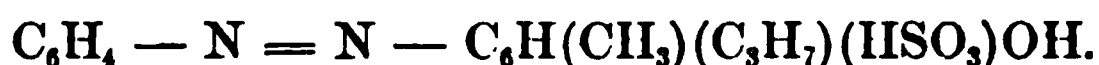
Owing to the small amount of substance under examination, a combustion was out of question, but to judge from the appearance of this body, I should immediately call it alpha thymole disulpho-acid.

## XXIX.—ON THE COMBINATION OF DIAZO COMPOUNDS WITH ALPHA THYMOLE SULPHO-ACID.

BY JAMES H. STEBBINS, JR.

In my last paper I hinted at the possibility of combining diazo compounds with alpha thymole sulpho-acid, and I will take the liberty, on this occasion, of describing my experiments in this direction.

Azobenzole alpha thymole sulpho-acid,



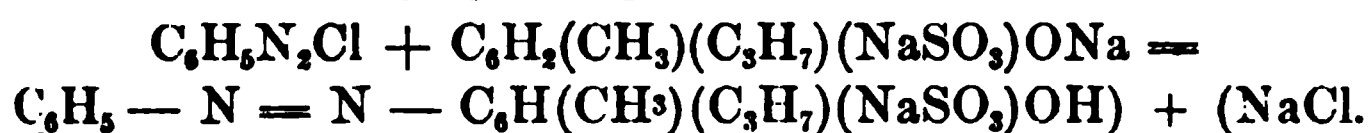
9.3 grms. aniline were dissolved in 22 grms. of strong  $\text{HCl}$ , diluted with 200 c.c. of water. To this mixture was then added, under constant stirring, 7 grms. of  $\text{NaNO}_2$ , previously dissolved in 50 c.c. of water.

The diazobenzole chloride thus formed was added gradually to an alkaline solution of the soda salt of alpha thymole sulpho-acid, consisting of 23.6 grms. of the sulpho acid and 8 grms. of  $\text{KOH}$ , previously dissolved in 200 c.c. of water. A copious yellow precipitate, composed of minute hair-fine needles, is thus formed, and constitutes the soda salt of our new dye-stuff.

This was then dissolved in boiling water and filtered from a black tarry fluid which was formed during the dissolving process. This

tarry fluid solidifies on cooling into a black mass, and evidently indicates that a decomposition of some kind had taken place.

The clear filtrate, when cold, deposits the soda salt of our new substance in the form of a network of minute, hair-fine, yellow needles. They were then collected on a filter, washed and dried at 100° C. When dry they take on a fine, metallic golden lustre, but their yellow color is changed to red-brown by the action of heat, which, however, does not destroy their tinctorial properties. This salt is easily soluble in water, and alcohol, and dyes wool of a pretty yellow color, in an acid bath. The formation of this substance may be explained, theoretically speaking, as follows:



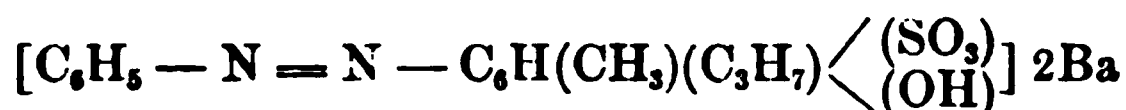
A combustion of the above gave:

THEORY.			
C <sub>16</sub> .....	192	.....	53.93 per cent.
H <sub>17</sub> .....	17	.....	4.77 “
N <sub>2</sub> .....	28	.....	7.86 “
S.....	32	.....	8.99 “
Na.....	23	.....	6.46 “
O <sub>4</sub> .....	64	.....	17.99 “
	<hr/> 356		<hr/> 100.00

FOUND.		
Carbon.....	53.77	per cent.
Hydrogen.....	4.72	“
Nitrogen.....	8.71	“

A sulphur estimation gave: 0.2060 grm. substance gave 0.1350 grm. BaSO<sub>4</sub>, which is equal to sulphur 8.99 per cent.

#### BARIUM SALT.



A part of the soda salt was dissolved in boiling water, and treated with an excess of BaCl<sub>2</sub>. This threw down the barium salt as an amorphous yellow precipitate. This was collected on a filter, washed several times, and then treated with boiling water.

As soon as the fluid cools, the barium salt crystallizes in hair-fine yellow needles. These are insoluble in cold, and only sparingly soluble in boiling water, but quite soluble in 90 per cent. alcohol.

A barium estimate was made, in order to ascertain the true molecular weight of the free acid, as well as to verify the assumed formula of the barium salt. 0.0902 grm. of substance gave 0.0238 grm. ash, or :

Barium ..... 18.35 per cent.

## THEORY.

Barium ..... 18.40 per cent.

This proves, beyond doubt, the correctness of the formula above set forth.

## FREE ACID.



A portion of the soda salt was dissolved in boiling water, and treated with an excess of strong HCl.

This threw down the free acid as a red amorphous precipitate, which, when dry, has a beautiful, golden-red, metallic lustre. The precipitate was then collected on a filter, and dissolved in boiling water. The hot solution so obtained was filtered, to separate it from a black resinous substance which seemed to be formed by the decomposition of the free acid while boiling with water. The filtrate, when cold, deposited the free dye-stuff, in the form of minute yellow, six-sided prisms. This body melts at  $215\frac{3}{4}^\circ \text{C}.$  with total decomposition. When heated on platinum foil, it burns with a red flame, and evolves copious yellow fumes, while on the foil is left a very difficultly combustible carbon residue. This interesting new dye-stuff, although of no commercial value, fully sustains the theory that we advanced in the beginning of this paper, namely, that diazo compounds can be made to combine with phenoles of the kind referred to.

The constitution of this compound can be explained from its reduction products. When treated with reducing agents, as zinc dust, or tin and HCl, the solution is decolorized, probably forming aniline, and amido thymole sulpho-acid. This latter, however, I have failed to isolate, as the quantity of material under examination was too small.

Parazotoluole alpha thymole sulpho-acid.



10.8 grms. of paratoluidine were dissolved in 22 grms. of strong HCl, diluted with 200 c.c. of water.

To this was then added, under constant stirring, 7 grms. of  $\text{NaNO}_2$ , previously dissolved in 50 c.c. of water.

The paradiazotoluole chloride thus formed, was allowed to rest for a while, and then mixed with 23.6 grms. of the soda salt of

- alpha thymole sulpho-acid, previously dissolved in 200 c.c. of water, made alkaline with 8 grms. of NaOH.

A copious yellow precipitate of the soda salt of parazotoluole alpha thymole sulpho-acid is formed. This was then collected on a filter, washed and dissolved in boiling water.

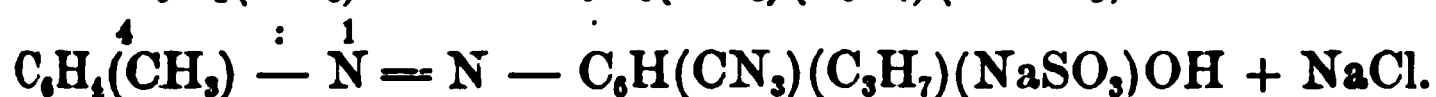
This compound is partially decomposed by boiling water, in the same manner as the previously described body.

The filtrate of the above when cold, deposits the soda salt in hair-fine, yellow-brown needles.

It is nearly insoluble in cold, but more soluble in boiling water.

It melts far above the boiling point of strong  $\text{H}_2\text{SO}_4$ .

The formation of this substance may be explained from the following reaction.



This compound is of particular interest, as it proves beyond a doubt that diazo compounds, derived from the higher homologues of aniline, can be combined in the same way as diazobenzole, with thymole sulpho-acid, to form compound azo dye-stuffs.

From the foregoing we might naturally be led to expect that diazo-xylol and diazo-naphthalene can, in like manner, be combined with alpha thymole sulpho-acid. The verification of this assumption will be detailed in the following experiment :

*Azoxylole alpha thymole sulpho-acid,*



This compound is produced by dissolving 6.1 grms. of xylydine in 11 grms. of strong HCl, diluted with 100 c.c. of water. To this is then added, under constant stirring, 4.2 grms. of  $\text{KNO}_3$ , previously dissolved in 50 c.c. of water.

The diazo-xylol chloride thus formed is poured, little by little, into a solution consisting of 17.3 grms. of the soda salt of alpha thymole sulpho-acid, dissolved in 100 c.c. of water, rendered alkaline with 4 grms. of KOH.

A copious brown precipitate of the soda salt is thus formed. This was then collected on a filter, washed several times, and then dissolved in boiling water. The solution so obtained was then filtered, in order to separate it from a tarry residue which was formed while dissolving with boiling water. The filtrate, when cold, coagulates into a thick, slimy mixture, and as this failed to crystallize, one-half of it was then treated with a concentrated solution of  $\text{BaCl}_2$ .



This immediately threw down a copious, yellow-brown precipitate of the barium salt. It was then washed several times with water, in order to remove all traces of  $\text{BaCl}_2$ , and then dissolved in boiling alcohol, and allowed to cool.

#### BARIUM SALT.



This salt is obtained, as already described, from the soda salt, by treating with  $\text{BaCl}_2$ .

It is insoluble in cold, and very nearly insoluble in boiling water. It is, however, quite soluble in hot 90 per cent. alcohol, from which it crystallizes in two forms. By rapid evaporation of the hot alcoholic solution, the barium salt is deposited, as a network of hair-fine, yellow needles. By slow evaporation, on the contrary, pretty lemon-yellow leaflets are obtained.

This salt should contain the following percentage of barium :

#### THEORY.

Barium . . . . . 15.92 per cent.

#### FOUND.

Barium . . . . . 15.42 per cent.

#### FREE ACID.



This compound was obtained by treating the second half of the slimy soda salt with an excess of strong  $\text{HCl}$ , which threw it down as a copious red-brown precipitate.

This was then collected on a filter, and washed with cold water till the excess of  $\text{HCl}$  had been removed, and was then treated with boiling water, in which it is but sparingly soluble.

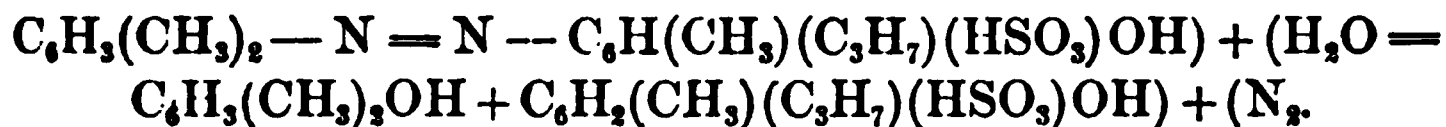
The hot solution was then filtered, in order to separate it from a tarry residue, and the filtrate allowed to cool. When cold, the free acid separates out in little, yellow, hair-fine needles, which were cooled on a filter, washed and dried.

Boiling water acts upon this compound in the same manner as the two previously described bodies ; that is, there is partial decomposition, with evolution of gas bubbles (probably nitrogen), and formation of a tarry residue which smells very much like xylenole.

Therefore, from what we have seen, the unstability of these compounds might lead us to assume that the azo radical ( $-\text{N} = \text{N}-$ ) is only very loosely bound to the benzole nucleus in thymole sulpho-acid, and that, consequently, the mere boiling with water is

sufficient to loosen the bonds between them, at the same time giving off nitrogen, and forming the corresponding phenole.

Should this be the case, the decomposition referred to might be expressed as follows:



Compounds of this nature might be looked upon as belonging to the diazo group; but, even were this assumed, I hardly think that the mere splitting up of the molecule (as above indicated) in these substances, is sufficient proof to classify them among the diazo compounds.

Diazo compounds are, as is well known, subject to a large number of transformations, of which the above reaction is one. But setting this one aside, I cannot find among my new substances any other characterizing features which should justify me in putting them under the head of diazo compounds; and I must, therefore, look upon them as highly unstable compound azo bodies.

It will likewise be observed here, that the solubility of these substances decreases as the molecular weight rises, a fact which might have a tendency to explain their increased instability.

Another curious fact may be mentioned here, namely, that these substances have all the same yellow shade of color, whereas the dye-stuffs derived from phenole and naphthole usually increase in depth of shade as the molecular weight rises.

For the above reason it was, therefore, not considered worth the while to try the action of diazonaphthalene on thymole sulpho-acid, as it might be supposed that the compound resulting from such a reaction would be too unstable to investigate, or that there might be no compound found whatsoever.

Therefore, to sum up, it will be seen that thymole sulpho-acid can be combined with diazo compounds, but that the resulting substances increase in instability as their molecular weight rises.

# THE AMERICAN CHEMICAL SOCIETY.

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## XXX.—PROCEEDINGS.

*Annual Meeting, Friday, Dec. 2, 1881.*

The meeting was called to order at 8:40 P. M., Prof. A. R. LEEDS in the chair.

The minutes of the previous meeting were read, and after correction, were approved.

The President had no report.

The Board of Directors had no report.

The Treasurer reports that we had a balance on hand at last annual report of \$342.62 ; received for dues, etc., for 1881, \$1,262.95 ; total, \$1,605.57 ; expenditures for 1881, \$1,270.31, thus leaving a balance on hand of \$335.26. From this, however, several due bills are to be paid, which, when paid, would leave in the treasury about \$275. On motion, the Treasurer's report was duly accepted.

The Librarian then stated that some books needed binding. The report was duly accepted.

The Curators then reported that the Society had been presented with a fine microscope.

Dr. LEEDS then stated on behalf of the Committee on the Endowment Fund, that the fund is progressing slowly. He mentioned a new contribution of \$50, and stated that the Fund is now approaching the sum of \$10,000.

After which the election of officers and committees was in order.

Dr. SQUIBB here stated his desire to have his name withdrawn from the list of Vice-Presidents.

After some remarks by Messrs. CASAMAJOR and NICHOLS, his withdrawal was accepted with regret.

After which the Chair appointed Messrs. CASAMAJOR and GROTHE to act as tellers in collecting the ballot. The following gentlemen were voted for

*For President.*

Profs. MORTON and MALLET having received the largest number of votes, it became necessary to elect one of these gentlemen, as there was not a majority of votes in either case.

Dr. SQUIBB moved that the Secretary cast an affirmative ballot for Prof. MALLET. This being seconded, was carried unanimously.

The Secretary then cast an affirmative ballot, thus electing Prof. J. W. MALLET as President.

*For Vice-Presidents.*

Mr. NICHOLS then moved that the tellers cast an affirmative ballot for the candidates having the largest number of votes.

It was then amended to lay this on the table. On motion, this was unanimously carried.

After which Mr. NICHOLS moved that his first motion be accepted. Duly carried.

The tellers then cast an affirmative vote for Messrs. LEEDS, WALLER, PRESCOTT, HABIRSHAW, GOESSMAN and LUPTON.

The rest of the ticket was duly elected.

After which Dr. ALSBERG stated his desire to resign. Some remarks and nominations by Messrs. NICHOLS, ELLIOTT and GEYER then followed, after which Dr. ALSBERG's resignation was accepted.

Mr. BENJAMIN then nominated Dr. SLOANE as Treasurer. It was moved that the tellers should cast an affirmative vote for Dr. SLOANE. This being seconded, was duly carried.

The tellers then declared Dr. SLOANE elected. Mr. BENJAMIN then nominated Mr. HALLOCK to take Dr. SLOANE's place on the Committee of Nominations. This was carried by acclamation.

It here became necessary to elect three more Directors, to fill vacancies caused by resignations.

The following gentlemen were then nominated: Messrs. MOORE, EDMER, RICKETTS, GEYER and HERRESHOFF.

It was here moved that the nominations be closed. Carried unanimously.

Dr. SQUIBB then moved that the meeting be adjourned till the second Friday of the month, in order that the papers announced might be read. On motion, this was duly carried.

Mr. ELLIOTT then mentioned the sudden death of Dr. JOHN LIPPS, and stated that only \$7 were found on his person, and with \$300 debts.

Mr. ELLIOTT then moved that a committee of four be appointed to look after and collect the necessary funds for his burial.

This motion was duly carried.

Mr. NICHOLS then made the following amendment: That Mr. ELLIOTT be appointed as a committee of one, and that he have power to add to his list at discretion. Carried.

The tellers then announced that Messrs. RICKETTS, HERRESHOFF and GEYER had been elected as Directors.

After which the meeting adjourned.

The total list of officers for 1882 is as follows:

*President,*

Prof. J. W. MALLET.

*Vice-Presidents,*

A. R. LEEDS,  
E. WALLER,  
A. B. PRESCOTT,

W. M. HABIRSHAW,  
CHAS. A. GOESSMAN,  
N. T. LUPTON.

*Corresponding Secretary,*

P. CASAMAJOR.

*Recording Secretary,*

JAMES H. STEBBINS, JR.

*Treasurer,*

T. O'C. SLOANE.

*Librarian,*

GEO. PROCHAZKA.

*Curators,*

A. G. ROSSI,      WM. RUPP,      A. A. FESQUET.

*Committee on Papers and Publications,*

ARNO BEHR,      A. R. LEDOUX,      H. ENDEMANN.

*Committee on Nominations,*

ARTHUR H. ELLIOTT,      J. P. BATTERSHALL,  
O. H. KRAUSE,      J. B. F. HERRESHOFF,  
A. E. HALLOCK.

*Directors,*

A. R. LEEDS,  
E. WALLER,  
JAMES H. STEBBINS, JR.,  
GEO. A. PROCHAZKA,  
C. F. CHANDLER,  
J. B. F. HERRESHOFF,

W. M. HABIRSHAW,  
P. CASAMAJOR,  
T. O'C. SLOANE,  
H. ENDEMANN,  
W. E. GEYER,  
H. MORTON,

P. DE P. RICKETTS.

JAMES H. STEBBINS, JR.,

*Recording Secretary.*

REPORT OF THE COMMITTEE ON THE ENDOWMENT FUND.

For the information of the members not intimately conversant with the business difficulties with which the foundation, in this country, of a national organization of chemists has been attended, it should be stated that the proposition to endow the publications of the Society, grew out of the great burden entailed by the attempt to publish a journal at frequent intervals. The design in so doing was to afford to all of the members an opportunity for the prompt publication of their original researches, and, in addition, with the aid of a corps of abstractors, to supply a summary of the most valuable matters published abroad. This entailed a great deal of labor upon the Editor and other members of the Publication Committee, and upon the gentlemen who assisted them. It resulted in a handsome volume of 621 pages for the year 1879, and a volume of 480 pages for the year 1880. That the volume for the past year has been of less size, has been due to the fact that the Society has not had sufficient money to pay for a larger one. But it has continued the JOURNAL with all the resources at its disposal, and will continue to do so, avoiding at the same time incurring any debt.

To make this JOURNAL as honorable and useful to the profession in this country as possible, and as it should be to comport with the dignity of a national organization of chemists, a larger annual income than that afforded by the annual dues, is imperatively necessary. The appeal has met with a favorable response from most of the manufacturing chemists thus far applied to, and we doubt not that the sum required could be speedily raised if the wants of the Society were made known to all. An endowment fund of this char-

acter, would guarantee the perpetuity of the publication of the JOURNAL and the Society itself, and though the past year has been one in which but a very limited amount of matter could be published, for the reason above given, success in procuring the moderate sum still required to complete the endowment, would usher in a period of far greater prosperity and usefulness than the Society has as yet enjoyed. The list of the subscribers is given below :

LIST OF SUBSCRIBERS TO THE ENDOWMENT FUND OF \$15,000 OF THE  
AMERICAN CHEMICAL SOCIETY.

The interest of this sum, it is thought, will cover the annual deficit arising from the expenses connected with the Publication of the Society. The collection of the amounts subscribed, is contingent upon the raising of the entire sum.

HAVEMEYER & ELDER.....		\$1,000 00
MATTHIESEN & WIECHERS.....		500 00
HELLER & MERZ.....		200 00
JOHN M. MAISCH.....		50 00
WM. H. NICHOLS.....		500 00
E. WALLER.....		100 00
E. R. SQUIBB.....		100 00
P. CASAMAJOR.....		100 00
A. R. LEEDS.....		100 00
JAS. H. STEBBINS, JR.....		50 00
C. F. CHANDLER.....		200 00
HENRY MORTON.....		100 00
PICKHARDT & KUTTROFF.....		500 00
Subscription at Anniversary Meeting, 1880.		
M. ALSBERG.....	\$25 00	
GEO. A. PROCHAZKA.....	10 00	
A. H. KRAUSE.....	10 00	
GIDEON E. MOORE.....	10 00	
H. N. VAUGHAN.....	10 00	65 00
SANITARY ENGINEER.....		50 00
TESSIÈ DU MOTAY.....		200 00
J. GOLDMARK.....		100 00
WM. WEIGHTMAN.....		500 00
ROSENGARTEN & SONS.....		100 00
HARRISON, HAVEMEYER & Co...		100 00
MARTIN KALBFLEISCH'S SONS..		500 00

JAS. MORGAN & Co.....	500 00
McKesson & Robbins.....	250 00
W. H. SCHIEFFELIN & Co.....	100 00
H. S. DESHON.....	50 00
J. L. & D. S. RIKER.....	100 00
DODGE & OLCOTT.....	100 00
B. F. LEEDS.....	50 00
PFIZER & Co.....	500 00
A. A. FESQUET.....	100 00
HECTOR C. HAVEMEYER.....	300 00
EIMER & AMEND.....	100 00
WHITALL & TATUM.....	100 00
HENRY BOWER.....	100 00
HARRISON BROS. & Co.....	100 00
ELMORE & RICHARDS.....	100 00
Manufacturing Chemists Association of the United States, through HENRY BOWER, Esq., Secretary.....	400 00

NOTE.—The Association likewise donated \$100 for the immediate needs of the Publication Committee of the Society.

F. W. DEVOE & Co.....	100 00
MARTIN E. WALDSTEIN.....	50 00
W. M. HABIRSHAW.....	100 00
EDWARD HILL.....	100 00
LANMAN & KEMP.....	100 00
PETER COOPER'S GLUE FACTORY	100 00
H. J. BAKER & BRO.....	100 00
A. B. ANSBACHER.....	50 00
D. F. TIEMAN & Co.....	100 00
WALDRON SHAPLEIGH.....	25 00

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\$8790 00

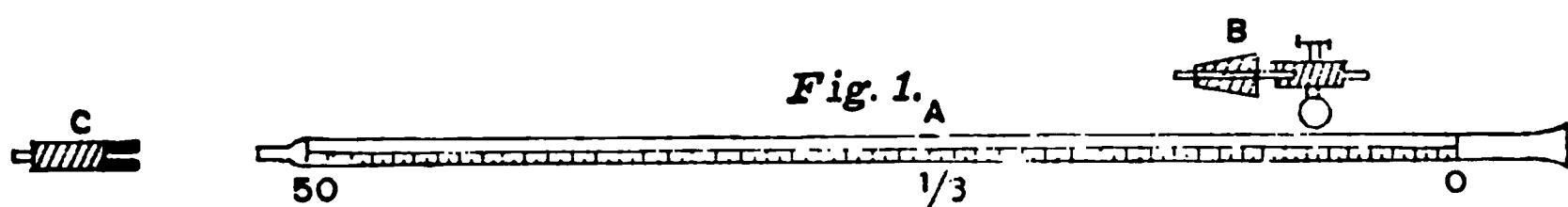
ALBERT R. LEEDS,  
JOHN M. MAISCH,  
JAMES H. STEBBINS, JR.,  
*Committee on Endowment Fund.*



# XXXI.—ON A MODIFICATION OF MOHR'S BURETTE ADAPTING IT FOR USE IN MEASURING CORROSIVE SOLUTIONS, ETC.

BY NELSON H. DARTON.

All who have frequent occasion to use the burette well know that the form contrived by F. Mohr is pre-eminently the most convenient, and that solutions may be delivered from it with less care, and, as I have found in a somewhat extended experience, greater accuracy than any other form. But as the solution of that much used reagent, permanganate of potassa, and some others, speedily destroys and is contaminated by the caoutchouc tubing attached, an instrument is required which will readily deliver the solution without having to come in contact with any organic bodies (this also applies to the delivery pipette). To accomplish this end, the Gay Lussac, Geisler, and Beck's burettes have been suggested, and extensively employed, but to me, at least, they have never given entire satisfaction. As I have had to use the burette so extensively myself, all the technical processes of my laboratory being executed by volumetric analysis, and I am now writing a work on that subject, that I perhaps have felt the need of a proper instrument more than the majority of dentists. So to obviate the difficulties in the above burettes, I have contrived a form which I have termed a modification, and after using it for some time with the most satisfactory results, I have come to the conclusion that it is perfect in all its applications. The following is a cut and description of this useful instrument:



The body, graduated as in the other forms, is drawn out and ground underneath to a perfectly plane surface, having the fine orifice in the centre; the stem should be about 15 m.m. long. While filling from above, holding a solution more than a half hour, etc., a small stopper *c* is used. It consists of a small piece of caoutchouc tubing, sufficiently large to fit over the stem tightly, and bearing a piece of glass bar half-way up its length, the upper end of which is ground to correspond with the under surface of the stem; this, when adjusted, forms an air-tight connection. The other end of the instrument is flared to receive a stopper, through which passes a glass

tube bearing a rubber tube, and pinch-cock, which is used to control the access of air through a small glass jet attached, thus allowing the solution to run out as required. The pipette is similarly constructed. The rubber tube connection shown in the cut as 5 c.m. long, is in practice as long as the burette, and the cock is thus placed at the hand of the operator as he is seated. In filling the burette, the jet is attached to the tube from the aspirator, the cork above the jet is opened, and a beaker full, or the required amount of solution in a beaker, placed under the stem; the aspirator cock is opened for a sufficient length of time, and the burette thus filled. In a very fine orifice burette, we may use two pinch-cocks, 5 c.m. or so apart, to control the access of air.

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### XXXII.—NEW FILTERING APPARATUS.

BY P. CASAMAJOR.

In the year 1875,\* I published descriptions of two funnels for filtering under pressure, one of which was to be applied to the method of filtration proposed by Dr. H. Carmichael, and described in the "Select Method of Chemical Analysis," of Mr. William Crooper (p. 429). In this method, the liquid is separated from the precipitate by the agency of a small disk of filtering paper, held against the perforated surface of a vessel, the interior of which communicates with an aspirator. This vessel, having the paper disk held against it, is placed directly in a platinum or porcelain dish, in which the precipitate is to be afterwards heated.

Dr. Carmichael made his vessel, communicating with the aspirator, of glass, but his method for making perforations on the flat side of this vessel was, to say the least, so very difficult, that very few chemists had succeeded in applying his method of filtration. The funnel which I used for the purpose, was of glass, the shape being that of a Platner's blow-pipe mouth-piece. The mouth of the funnel was closed by a small disk of filter paper, resting on a perforated platinum plate. This plate was also circular and slightly smaller than the disk of filtering paper. Both the perforated plate and disk of filter paper were held tightly against the funnel by the suction of an aspirator.

In the same paper there is a description of another funnel, to be used with the same filter, but, in using this funnel, whose shape is

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\* *American Chemist*, 5, 440, and *Chemical News*, 32, 46.

that of a large thistle tube, the perforated platinum plate is placed on the bottom of the funnel, and, over it, the small disk of filter paper, the edges of which slightly overlap those of the perforated plate. The liquid to be filtered is poured in the funnel. With this funnel, any ordinary aspirator may be used, but I have always used, in connection with it, a simple aspirator consisting of a straight vertical tube of small diameter, attached to the bottom of the funnel. I again called attention to this particular form of aspirator in a subsequent paper,\* published shortly after, in which are given fuller details as to its use.

The vertical tube acts by the weight of the column of water, which it holds suspended below the liquid. Its use was made possible by the fact that, when there was no more liquid above the moist disk of filter paper, this became impervious to air, and the column of liquid in the vertical tube continued to be held in suspension, but any additional liquid poured in the funnel went through the paper disk without any difficulty.

I pointed out that, instead of using a disk of filter paper, paper pulp or asbestos pulp could be poured into the funnel. The excess of water would run out, and a layer of paper or of asbestos would be left on top of the perforated plate, and around its edges, and form a very efficient filtering medium.

This aspirator, consisting of a vertical tube, was found so simple and convenient, that I tried to apply it to the funnel first described, which is used in Dr. Carmichael's system of filtration, but the experiments were not successful. In funnels of this shape, the perforated plate and disk of filter paper are held on the under side of the funnel by the suction of an aspirator. Whenever the aspirator ceases to act, the platinum plate and sheet of paper drop down.

The difficulty experienced was due to this: that the paper filter cannot be held in position unless there is a volume of liquid in the vertical tube, while, at the same time, the vertical tube cannot be filled unless the paper filter is held tightly against the funnel.

These are the antecedents of the filter and the aspirator which I now propose to describe.

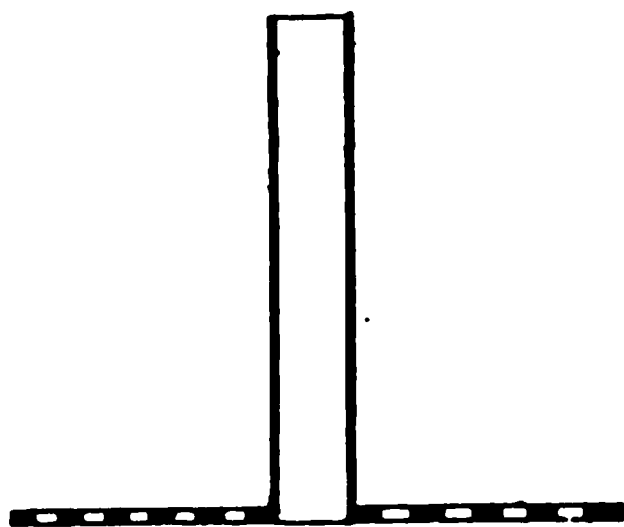
In this new filtering apparatus, the filtering medium is laid on a perforated plate, provided with a tube open at both ends, which is

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\* Funnels with auxiliary vertical tube; see *American Chemist*, 6, 124, and *Chemical News*, 32, 184.

firmly attached to the plate, over a hole of the same size as the tube. Figure 1 shows a section of the plate, with the tube attached.

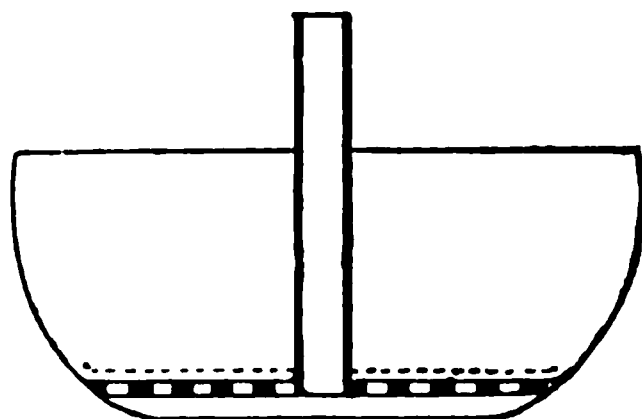
*Fig. 1.*



This perforated plate is laid on the bottom of a dish or crucible made of platinum, porcelain, or any other suitable material. The upper portion of the tube is connected with an aspirator, and there must be a small space left between the under surface of the plate and the bottom of the vessel, to allow the filtered liquid to pass through the filtering medium. The liquid which passes into the space under the plate, is removed through the tube by the action of the aspirator.

Figure 2 shows the perforated plate in position at the bottom of a platinum or porcelain dish. The filtering medium rests on top of the perforated plate, and is indicated by a fine dotted line. If the bottom of the vessel should be perfectly flat, it would be necessary to make the perforated plate slightly curved, with the concavity turned downward, to allow a space between the plate and the bottom of the vessel.

*Fig. 2.*



The filtering medium may be a piece of filter paper, or it may be deposited in the form of paper pulp or asbestos pulp, as already mentioned. If a paper filter is used, there should be a hole in it, to let the tube go through. The perforations in the plate should begin at a certain distance from the tube, so that every portion of the perforated surface may be covered with paper.

It is a very simple matter to make this perforated plate with its tube. Any jeweler can make a tube from platinum foil, and solder the joint with coin gold, which is sufficiently infusible for most purposes, and the tube can be soldered to the plate with the same material. This is the readiest way, and it is next to impossible in this country to have work of this kind done entirely of platinum.

We may also form this piece of apparatus in two portions. Any metal-spinner can turn, on a platinum plate, a tube 5 or 6 millimeters long, and, over this short tube, a platinum tube about 3 centimeters long, can be firmly placed. It is very important that the long plati-

num tube should come down as low as possible over the shorter tube, so that the pulp of paper or asbestos, may be deposited over the joint.

It is almost useless to mention that the weight of the perforated plate should be taken as part of the tare, with the weight of the platinum vessel, and of the asbestos, when a pulp of this material is used. The perforated plate remains in the crucible or dish, while the precipitate is heated, and it is afterward placed on the balance along with the precipitate. When asbestos is used, the requisite quantity should be placed in the vessel in which the precipitate is to be heated, the perforated plate should be added, and the whole sufficiently heated. The vessel and all the contents should then be placed on the balance, so as to obtain the total tare. If the platinum tube is not soldered to the perforated plate, it may with great convenience be left out of the count, as it may be easily removed by holding down the perforated plate with a spatula, and pulling off the tubes after the filtration is over. The quantity of asbestos required is very slight. Dry asbestos, weighing 1 decigram, if sufficiently fine, can easily cover a perforated plate with a surface of 6 square centimeters (about 1 square inch).

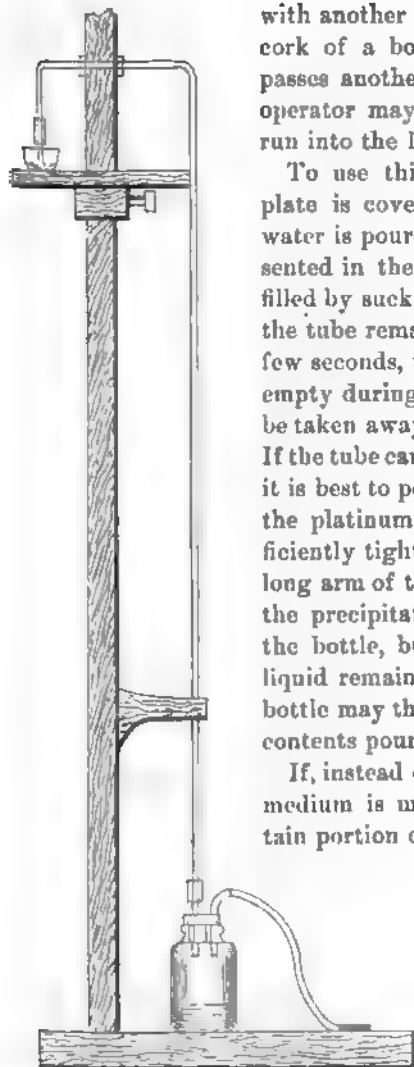
Very full details relating to asbestos filters may be found in an interesting paper of Mr. F. A. Gooch, read before the American Academy of Science, Feb. 13th, 1878, and published in the *Chemical News*, 37, 181. The author does not seem to be aware that I had proposed the use of asbestos pulp, in 1875, as mentioned above, by pouring the pulp over a perforated plate.

In the arrangement introduced by Mr. Gooch, the filtered liquid is forced through asbestos, lying on the perforated bottom of a crucible, by the action of an aspirator. There is a tight joint formed around the crucible, by forcing it into a large rubber tube, which also fits tightly on the top of a glass funnel, in the manner proposed for porous earthenware cones by Prof. Munroe. There is no doubt that a very good aspirator, for this filtering apparatus, would be a straight glass tube having a small diameter, connected by a rubber tube with the stem of the glass funnel.

With the new form of filtering apparatus, having a perforated platinum plate, with tube attached, any form of aspirator may be used, and there is no difficulty in using a vertical tube having a small diameter, like those already mentioned, in which the suction is caused by the weight of a column of the filtered liquid held in suspension. This aspirator with filter, shown in section, is represented in Figure 3. The aspirator tube is bent twice at its upper end, and there

terminates in a short vertical tube about 3 centimeters long, connected with the platinum tube, attached to the perforated plate, by a rubber tube. The long vertical portion of this tube is connect-

*Fig. 3.*



ed, at its lower end, by a short rubber tube, with another glass tube passing through the cork of a bottle. Through the same cork passes another tube, by means of which the operator may start the liquid, and make it run into the long vertical tube.

To use this apparatus, if the perforated plate is covered with filter paper, distilled water is poured in the platinum dish, represented in the figure, and the vertical tube is filled by sucking air from the bottle. When the tube remains filled with liquid, for even a few seconds, there is no fear of its becoming empty during the filtration. The bottle may be taken away and a beaker glass substituted. If the tube cannot be made to retain the liquid, it is best to pour some of the precipitate in the platinum dish, and this will make a sufficiently tight joint to keep the liquid in the long arm of the tube. By doing this, some of the precipitate may at first be carried into the bottle, but very soon nothing, but clear liquid remains in the glass tube. The glass bottle may then be taken away, and its liquid contents poured back into the platinum dish.

If, instead of a sheet of paper, the filtering medium is made from asbestos pulp, a certain portion of the pulp will inevitably pass

through the glass tube at first. This will have to be poured back into the platinum dish, even if no portion of precipitate has gone through. If paper pulp is used, and only a small por-

tion passes through, without any of the precipitate, there is no necessity of pouring it back.

After the tube has once remained permanently filled with the filtered liquid, no further difficulty will be experienced. The rest of

the liquid to be filtered may be gradually poured in the platinum dish, and subsequently hot water is added to wash the precipitate.

After the operation is completed, if the platinum tube is slipped from the rubber tube which connects it with the aspirator, the water held in the platinum tube will fall back in the crucible. This is easily got rid of by subsequent evaporation, but this quantity of water, and that which remains below the platinum plate, may be mostly carried off through the aspirator by carefully removing the precipitate from a point on the edge of the paper disk, and lifting this up with the point of a needle just sufficiently to let air go in, to clear the aspirator tube from liquid.

### XXXIII.—A METHOD FOR THE ANALYSIS OF MUSTARD.

BY ALBERT R. LEEDS AND EDGAR EVERHART.

During the past summer a large number of analyses of various articles of food and drink was made by the authors, on behalf of the State of New Jersey. Among the articles analyzed were thirty different kinds of mustards, as sold in shops. The method of analysis followed was that given in all text-books on the subject, and especially recommended by Blythe and Hassall. The process consists, as is well known, in estimating the moisture by drying at 100–110°; in determining the ash for the detection of mineral adulteration, and weighing the oil. The other adulterations are determined only qualitatively.

In this method reliance is placed principally on the estimation of the oil, and from this is calculated the amount of mustard supposed to be present in the mixture. Blythe gives the following formulæ for calculating the amount of mustard in a mixture of flour and mustard:  $x$  = amount of mustard and  $y$  amount of oil found,

$$\frac{33.9x}{100} + \frac{1.2(100 - x)}{100} = y \text{ and } \frac{36.7x}{100} + \frac{2(100 - x)}{100} = y.$$

This method of analysis and calculation might do very well if no foreign fat were added, or if none of the original mustard oil were extracted. In fact, however, flour baked in oil or fat is frequently added to a mustard from which a portion of the original oil has been extracted, and furthermore, mustard, before being put on the market, is subjected to hydraulic pressure and loses from 12 to 20 per cent. of its oil. From this method of analysis, it is evident that satisfactory or reliable results cannot be obtained.



So far as is known, there has been only one attempt made to analyze mustard by its separate constituents. Hassall, in his book on "Food, its Adulterations and the Method for their Detection," proposes an analysis, partly direct and partly indirect. His method, and the results obtained by it, are those most quoted both in English and foreign books and journals.

His plan of analysis is as follows: The moisture and ash are determined as ordinarily, and the oil by extracting with ether. The myronate of potash is estimated by taking advantage of its well-known reaction with the myrosine contained in the mustard flour, in presence of water. Forty or fifty grains of the mustard are allowed to digest for twenty-four hours, with about 250 c.c. of water in a well-corked flask. At the end of that time all the myronate of potash will have been decomposed by the ferment myrosine into glucose, sulphate of potash, and mustard oil (allylthiocarbamide), according to the equation  $C_{10}H_{18}KNS_2O_{10} = C_6H_{12}O_6 + KHSO_4 + C_4H_5NS$ . The contents of the flask are distilled, and all of the allylthiocarbamide goes over with the water vapor. The end of the condenser dips below the surface of some strong ammonia water to prevent loss of the volatile oil. When no more oily drops come over with the distillate, the receiver and its contents are removed and allowed to stand until the allylthiocarbamide has combined with the ammonia, forming thiosinamine ( $C_4H_5NS.NH_3$ ). The solution is evaporated to dryness in a tared platinum dish, and from the amount of thiosinamine found, is calculated the myronate of potash.

So far the method is all that can be desired, but the remaining part is open to very serious objections. For the determination of the myrosine and sulphocyanide of sinapine, a combustion of the mustard is made with soda-lime for the total nitrogen, and another portion is fused with alkaline carbonates and nitrates, to estimate the total sulphur. As much nitrogen and sulphur as is contained in the myronate of potash, is subtracted from the total nitrogen and sulphur, and from the two residues are calculated the amounts of myrosine and sulphocyanide of sinapine. The cellulose is estimated by difference.

The whole of the sulphur residue, and so much of the nitrogen as is necessary, are calculated into sulphocyanide of sinapine, and the remaining nitrogen into myrosine. But as myrosine contains about 1.6 per cent. of sulphur, further calculations are necessary. Such calculations may or may not yield correct results, for one has as much right to calculate all the remaining sulphur first into myrosine, or all the nitrogen into either myrosine or sulphocyanide of sinapine, as the



sulphur into the last-named compound. The results cannot be calculated algebraically because there is only one equation, and this equation has two unknown factors. In one of the samples of mustard analyzed by Hassall he finds myrosine 31.686 per cent., sulphocyanide of sinapine 5.714 per cent. Taking his percentages of nitrogen and sulphur, and calculating the total nitrogen first into myrosine, one finds, myrosine 31.43 per cent., sulphocyanide of sinapine 3.95 per cent.

For the reasons above detailed, the following attempt was made to work out a method for the analysis of mustard, which should be direct throughout, and should rest on an actual separation and estimation of the several constituents. The moisture and ash are determined as usual. The mustard oil is extracted with ether in the following manner: A weighed portion of mustard, after drying at  $105^{\circ}$ , is carefully brushed into a plaited filter. The filter and its contents are placed in a funnel with straight sides. The stem of the funnel is connected, by means of a well-fitting cork, with a small tared flask partially filled with ether, while the funnel itself is connected with an upright condenser. On cautiously boiling the ether, its vapor is constantly condensed, and, falling on the mustard, extracts the oil which is retained in the flask. When all of the oil is removed, the ether is distilled off, and the flask and contents, after drying at  $100^{\circ}$ , re-weighed. The difference between the weight of the flask alone, and that of the flask and oil, gives the amount of oil. After the ether has evaporated from the mustard residue, a tared flask, containing half water and half alcohol, is substituted for that containing ether, and the contents are boiled and condensed as before. The dilute alcohol dissolves both the sulphocyanide of sinapine and the myronate of potash, while it coagulates the myrosine and leaves both it and the cellulose undissolved. After all the sulphocyanide of sinapine and myronate of potash have been extracted, the contents of the flask are rinsed into a tared platinum dish, evaporated to dryness, dried at  $105^{\circ}$ , and weighed. The dish and contents are then ignited and weighed. The difference of weight before and after ignition, gives the total amount of sulphocyanide of sinapine and myronate of potash. Subtracting the amount of the latter, the difference is the amount of sulphocyanide of sinapine.

After the extraction with alcohol, the filter contains only the myrosine and cellulose, together with a little coloring matter. The alcohol is allowed to evaporate spontaneously, and then the myrosine and cellulose are treated in the cold with a one-half per cent. soda

solution. The solution containing the myrosine is decanted through a weighed filter, and the residue is treated again in the same manner. By this treatment all the myrosine is obtained in solution. The cellulose on the filter is dried, weighed, ignited, and the ash weighed. The difference between the two weights gives the cellulose.

The solution containing the myrosine is just neutralized with dilute hydrochloric acid, and about 50 c.c. of Ritthausen's cupric sulphate solution added. The solution is then *exactly* neutralized with dilute soda, and the heavy green precipitate of the compound of copper and myrosine allowed to settle to the bottom of the beaker.

The precipitate is collected on a weighed filter and dried at 110°. After drying, the weight of the precipitate is taken. It is then ignited, and the ash weighed, the difference giving the total amount of myrosine.

The two following analyses of a sample of Brown Mustard Farina, prepared by H. K. & F. B. Thurber & Co., New York, were made simultaneously. Afterwards a third analysis was made on the same sample.

● ANALYSIS OF BROWN MUSTARD FARINA.

	1	2	3
Moisture . . . . .	6.78	6.90	6.82
Myronate of potash . . . . .	0.61	0.61	0.72
Sulphocyanide of sinapine . . . . .	10.97	11.19	11.21
Myrosine . . . . .	28.45	28.70	28.30
Mustard oil . . . . .	29.22	29.21	29.19
Cellulose by difference . . . . .	20.24	19.55	20.06
Ash . . . . .	3.73	3.84	3.70
	<hr/>	<hr/>	<hr/>
	100.00	100.00	100.00

A combustion was made of the same mustard, and the nitrogen determined. The sulphur was likewise determined by fusing with alkaline carbonates and nitrates, and precipitating with barium chloride.

Nitrogen = 5.337 per cent. Sulphur = 1.489 per cent. Calculating the amounts of nitrogen and sulphur in the myronate of potash, the sulphocyanide of sinapine and the myrosine, we find nitrogen, 5.342 per cent., and sulphur, 1.50 per cent.

If the amounts of the three last named constituents of the mustard be deduced from the total amounts of nitrogen and sulphur according to Hassall, the following will be the percentages :

Myronate of potash . . . . .	0.61 per cent.
Sulphocyanide of sinapine . . . . .	10.71 “
Myrosine . . . . .	28.52 “

In this case, the results obtained by calculation from the percentages of nitrogen and sulphur, are almost identical with those obtained by direct determinations. But the greater ease and certainty of the direct method, and the very considerable errors which are possible when calculations are instituted upon results differing but very slightly from the true ones, must strongly recommend, it appears to us, the direct as compared with the indirect method.

If the mustard is adulterated with starch or flour, the foregoing scheme of analysis may be used as well in the case of pure mustard. After the extraction of the oil with ether, and the sulphocyanide of sinapine and myronate of potash with alcohol, the residue may be treated either with malt extract or with acids under pressure to convert the starch into glucose. The glucose may be estimated as usual.

Fearing that starch was not entirely insoluble in dilute alcohol, and not being able to learn anything on the subject from books, an experiment was made of boiling starch for some time in a mixture of half alcohol and half water. The boiling liquid was filtered hot, and to the filtrate a few drops of iodine solution added. No blue coloration taking place, the starch may be considered insoluble in dilute alcohol, and hence could not interfere in the determination of the sulphocyanide of sinapine and myronate of potash.

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#### XXXIV.—UPON THE COMPOUNDS OF THE AROMATIC BASES WITH METALLIC SALTS, WITH A NOTE UPON THIOCARBANILIDE.

BY ALBERT R. LEEDS.

##### *Introduction.*

The author was led to a study of these compounds by the following considerations :—

I. The energy, in many cases attended by the evolution of much heat, with which a number of them are formed.

II. The hope that a study of the structural formulæ of compounds of so considerable a degree of complexity, and containing both metallic and non-metallic basic radicals in combination with acid radicals, might be attended with theoretic interest.

III. The expectation, which, indeed, first suggested the inquiry, that by removal or replacement of the metallic element in these compounds by reactions, induced by organic compounds of sulphur and related substances, these metallo-aromatic bodies would serve as material in the institution of an interesting line of research. This

expectation was in part verified, such derivatives being obtained either by laboratory operations, conducted as usual, or with the aid of sun-light, but it was then found that they had already been prepared by simpler and more direct methods. These remarks apply more especially to thiocarbanilide and related compounds.

But it was found that the great majority of these compounds were quite unstable, undergoing decomposition merely by treatment with water. Consequently, the attempts to form them by double decomposition of aqueous solutions of their constituent salts were mainly unsuccessful. Moreover, the double salt of the aromatic base, containing the same acid radical in combination with both base and metal, could not be formed. This result is in opposition to that obtained by Schiff, not only in the case of antimonious chloride, but also in regard to other compounds. Finally, if the combinations are to be regarded as either amines or ammoniums, in which a portion of the hydrogen is replaced by a metal, and the radical thus formed enters into combination with either the acid itself or the acid radical, then it might be anticipated that such a compound would again unite with platinic chloride to form a double salt. Numerous attempts were made to form such double salts, but they were unsuccessful, the metallic salt dropping out, and a double chloride of platinum and the aromatic base alone remaining.

In formulating them, therefore, as ammoniums, which is the simplest mode of explaining their composition, these points of weakness in the experimental data must be kept in mind. And whilst the following compounds might receive a nomenclature in accordance with the theory of their being metallo-ammoniums, the antimony compound being the chloride of triphenylstibonium, the salt of mercury being the chloride of diphenylmercurammonium, and so on, yet I have seldom used these terms, not only because of their cumbersome character, but also because they necessarily assume what as yet is not very satisfactorily proven.

*Authorities consulted.*

H. SCHIFF : Upon the Anilo-metallic Compounds and upon the Formation of Fuchsine. C. R. 56, 268.

H. SCHIFF : Researches upon the Mercuraniles, C. R., 56, 491.

“ “ Researches upon the Trimetalaniles, C. R., 56, 1095.

C. FOSTER : Upon Mercuridphenylammonchlorür,  $\text{NC}_6\text{H}_5\cdot\text{Hg}\cdot\text{HCl}$ . *Ber. der Deutsch. Chem. Gesell.*, 7, 294.

O. KLEIN : Upon the Combinations of Organic Bases with Mercuric Chloride. *Ber. der Deutsch. Chem. Gesell.*, 11, 743 and 1741.

LIPPMANN and VORTMANN: Upon a Combination of Cobaltous Chloride with Aniline. *Ber. der Deutsch. Chem. Gesell.*, **II**, 1069.

Watts' Dictionary of Chemistry.

Beilstein's Lehrbuch der Organischen Chemie, pp. 875 and 936.

### *Compounds with Aniline.*

#### I. ALUMINA SALTS.

*Aluminic chloride.*—Three molecules of aniline were heated with one of aluminum chloride. The resulting compound was washed with alcohol, water and ammonium chloride, in none of which it appeared to dissolve. After washing out all the excess of aniline with alcohol, a white crystalline powder remained.

The operation was repeated, alcohol only being used to dissolve the resultant compound. The crystals which separated out of the first portions of the alcohol, proved to be chloride of aniline. The white powder which remained after repeated treatment with boiling alcohol, was alumina. In other words, no compound of aniline with aluminic chloride is formed when the former is heated with  $\text{Al}_2\text{Cl}_6$ ,  $6\text{H}_2\text{O}$ . The latter is decomposed into alumina and hydrochloric acid, and merely a hydrochlorate of aniline is formed.

*Aluminic sulphate.*—On mixing the two bodies, only a slight elevation of temperature ensued, and the heat was afterwards raised to the boiling point of aniline. The excess of aniline was washed out by repeated decantation with boiling alcohol, in which it is not very soluble, and a portion of the compound crystallized from the same solvent. It crystallized, apparently, in small white needles. On heating, these crystals blackened, and burnt with a small flame. Their analysis gave only 4.4 per cent. of alumina. The compound  $3\text{C}_6\text{H}_7\text{N}.\text{Al}_2(\text{SO}_4)_3$ , requires 8.84 per cent. of alumina. They were not a compound with aniline, but some of the undecomposed sulphate together with aniline which had not been perfectly removed by repeated washing.

*Aluminic hydrate.*—Neither this nor any other salt of alumina entered into combination.

#### II. ANTIMONY.

*Antimonious chloride*,  $3\text{C}_6\text{H}_7\text{N}.\text{SbCl}_3$ .—First formed by adding 3 molecules of aniline to one of antimonious chloride and heating. The resulting compound had a pinkish color, probably due to exposure to the air. It lost this color after washing with alcohol, and became perfectly white. It was decomposed by water.

Schiff prepared this substance as above, and also by adding aniline to a solution of antimonious chloride in benzine. This yielded a white crystalline mass, which was soluble in aniline only, and crystallized out of the latter in slender needles. According to Schiff, it is converted into a double salt by hydrochloric acid, melts at  $80^{\circ}$ , solidifies again in long needles, and is partially decomposed by distillation (Watt's Dictionary, 4, 474).

These statements were not verified. On the addition of hydrochloric acid the compound was decomposed, hydrochlorate of aniline crystallizing out of the solution. It does not melt at  $80^{\circ}$ , nor even at  $240^{\circ}$ , being in part decomposed and partially subliming. This was true both when heated in the air, and when the attempt was made to distil it in vacuo.

When the same compound was made by mixing antimonious chloride with aniline, the temperature rose from  $23^{\circ}.5$  to  $82^{\circ}.5$ . It was washed repeatedly with alcohol, in which it is but slightly soluble, and a portion recrystallized from the same. It formed minute needle-shaped crystals. It was decomposed by water, the solution giving the reaction for antimony and aniline.

*Antimonic chloride*,  $5C_6H_7N.SbCl_5$ .—Formed in like manner as the preceding compound, which it closely resembles in appearance and properties. The temperature rose from  $22^{\circ}.5$  to  $85^{\circ}$ .

*Antimonious iodide*,  $3C_6H_7N.SbI_3$ .—"To obtain the combination of antimonious iodide with aniline, it is necessary to raise the temperature to  $100^{\circ}$  or  $120^{\circ}$ . If an excess of aniline is used, the compound is formed in little yellow needles, colored undoubtedly by a small quantity of iodine. By distillation a large portion is decomposed" (Schiff).

I did not repeat the experiment.

*Antimonious sulphide*.—Does not enter into combination.

### III. ARSENIC.

*Arsenious chloride*,  $3C_6H_7N.AsCl_3$ .—On the addition of 3 molecules of aniline to one of arsenious chloride, an energetic combination ensued, the temperature being considerably raised, and the mixture solidifying afterwards to a whitish crystalline mass. This was purified by recrystallizing three times from alcohol. The final product was in small white crystals, not well defined and becoming red by exposure to the air.

According to Schiff (*loc. cit.*), this compound melts at about  $90^{\circ}$ , distils without decomposition between  $205^{\circ}$  and  $210^{\circ}$ , and is some-

what soluble in water, with separation of arsenious acid. We sublimed it in vacuo, at circa  $205^{\circ}$ , at which temperature it sublimes without previous melting and with partial decomposition. The product lost the red tint of the original substance, and was in the form of minute white plates, polarizing finely. It was decomposed by hydrochloric acid, and after slow evaporation of the hydrochloric acid solution, only aniline hydrochlorate remained, the  $\text{AsCl}_3$  having volatilized.

*Arsenious iodide*,  $3\text{C}_6\text{H}_7\text{N}.\text{AsI}_3$ .—"It forms at an elevated temperature, and is decomposed neither by cold water nor by dilute hydrochloric acid. It is slightly soluble in benzine and in cold alcohol. Boiling alcohol decomposes it in a curious manner; brown flocks of monoiodide of arsenic are obtained, and the alcohol on evaporation leaves long needles of hydriodide of iodoaniline" (Schiff).



The experiments were not repeated.

*Arsenious sulphide*.—Does not enter into combination.

#### IV. BARIUM.

No combination ensued when aniline was subjected to the action of barium acetate, chloride, iodide, nitrate and sulphide.

On heating aniline with barium chloride or iodide, dissolving in boiling ammonium chloride, and crystallizing, small white needles were formed. On the addition of water, these went into solution very readily. After washing with alcohol, they were found to consist only of unchanged barium chloride or iodide. When the mass obtained by heating aniline with the barium salt was treated with alcohol, no compound could be crystallized out.

#### V. BISMUTH.

$3\text{C}_6\text{H}_7\text{N}.\text{BiCl}_3$ .—According to Schiff (*loc. cit.*), this is a fusible, indistinctly crystalline mass, which is very slowly decomposed by water, and turns violet when heated. The experiment was not repeated. I found that the nitrate formed no compound.

#### VI. CADMIUM.

*Cadmium chloride*,  $2\text{C}_6\text{H}_7\text{N}.\text{CdCl}_2$ .—This and the corresponding compounds of cadmium bromide and iodide, were obtained by heating with excess of aniline. On boiling all went into solution, but after cooling the entire mass crystallized in a solid lump. The excess of aniline was washed out with alcohol, in which these cad-



mium compounds are insoluble. Neither could they be crystallized from chloroform, ether, or water. Ammonia exercised a solvent action, but appeared at the same time to effect some decomposition. The three compounds were boiled with calcium chloride, in which they were readily soluble, and from which they crystallized out in fine white needles. The  $2C_6H_7N.CdCl_2$  was purified from  $CaCl_2$  by redissolving in water, from which it crystallized in very minute, flexible needles, of a white color, with silky luster. These needles readily compacted themselves into soft, tufted masses.

As crystallized from ammonium chloride, all the compounds formed by aniline with cadmium or other salts were not pure. It was found to be the best method, in almost all instances, to heat the salt directly with the aniline, and, after getting rid of the excess of the latter, to crystallize repeatedly out of alcohol. When the cadmium chloride was so made, the temperature rose from  $18^\circ$  to  $42^\circ$ .

*Cadmium bromide*,  $2C_6H_7N.CdBr_2$ , as crystallized from calcium chloride, formed longer needles, which aggregated in little tufts and had a satin luster.

*Cadmium iodide*,  $2C_6H_7N.CdI_2$ .—This compound, when recrystallized from boiling ammonium chloride, formed like the two former when crystallized from the same solvent, long thick white needles. The needles were flat, and of a beautiful pearly luster. On exposure to light and air their color turned to a faint pink.

*Cadmium nitrate*,  $2C_6H_7N.Cd(NO_3)_2$ .—It was obtained by boiling cadmium nitrate with excess of aniline, and washing out the latter with alcohol. It crystallizes in white needles, aggregating into lamellar masses, with a silky luster.

## VII. CALCIUM.

*Calcium chloride*,  $2C_6H_7N.CaCl_2$ .—On mixing aniline with calcium chloride, the temperature rose from  $18^\circ$  to  $20^\circ$ . The mixture was then raised to the boiling point of the aniline, when, on cooling, a hard crystalline mass was formed. This was drained from the excess of aniline, pulverized, and dried between filter paper. On account of the extreme solubility of the compound in alcohol, it was washed only once in this solvent, and then dissolved and crystallized. It formed long, stout prisms, perfectly colorless, and of a glacial appearance.

$2C_6H_7N.CaI_2$ .—The temperature of the mixture rose from  $20^\circ.5$  to  $24^\circ$ . It behaved exactly like the corresponding chloride, forming crystals similar to those above described.



Experiments made with a view of crystallizing out the compounds from boiling ammonium chloride, were not satisfactory, owing to their great solubility both in water and alcohol.

#### VIII. CHROMIUM.

Neither chromic chloride or sulphate entered into combination.

#### IX. COPPER.

When cupric acetate is added to aniline, the mixture immediately becomes black, and solidifies to a very hard mass. In order to insure complete union this mass was broken up and triturated in a mortar with aniline. After washing several times with alcohol and boiling it with the same, there was left a black, uncrystallizable mass. When dissolved in hydrochloric acid this substance gave no reaction for aniline.

*Cuprous chloride and cupric nitrate.*—Similar black substances were produced, and no certain evidence was obtained that definite compounds with these salts were formed, or if formed, were stable in contact with air or solvents. The same results were true of cupric sulphate.

#### X. COBALT.

*Cobaltous chloride,  $2C_6H_7N \cdot CoCl_2$ .*—When the cobaltous chloride and aniline were mixed together, the temperature of the mixture rose from  $18^{\circ}.5$  to  $104^{\circ}.5$ . The resulting compound was washed several times with alcohol. Finally one portion was recrystallized from absolute, the other from common alcohol. Both formed very beautiful red crystals, the former in rhomboidal plates, the latter in prismatic needles. When exposed to the air, these crystals assume a cobalt blue color.

The constitution of these bodies has already been investigated by Lippmann and Vortmann, who formed the first substance as a pale red precipitate by the addition of an alcoholic solution of cobaltous chloride to aniline. They found its composition to be  $2C_6H_7N \cdot CoCl_2 + 2C_2H_6O$ . The blue compound is derived from the former, by spontaneous decomposition in the atmosphere, forming  $2C_6H_7N \cdot CoCl_2$ .

We found, as had already been shown by the authors above-mentioned, that these compounds are decomposed into their constituents on solution in water. Also that on treatment with an alcoholic solution of platinic chloride, a double salt with aniline, not containing in combination the cobaltous chloride, is formed.

*Cobaltous nitrate and sulphate.*—In aqueous or alcoholic solutions, or when heated directly with aniline, these salts did not combine.

#### XI. IRON.

*Ferric chloride.*—On adding ferric chloride to aniline, so energetic a reaction ensued that the temperature of the mixture rose to  $80^{\circ}$ . A black mass was formed, incompletely soluble in alcohol and water, and which could neither be sublimed nor crystallized from any solvent which was employed.

*Ferrous chloride*,  $2C_6H_7N.FeCl_2$ .—On adding aniline to ferrous chloride the temperature is raised from  $21^{\circ}$  to  $23^{\circ}$ . It solidifies to a hard yellowish mass. After removing the excess of aniline by washing with alcohol, and dissolving in alcohol, the compound crystallizes in yellowish needles.

*Ferrous sulphate.*—It does not enter into combination.

#### XII. LEAD.

None of the salts of lead form compounds with aniline.

#### XIII. MAGNESIA.

The same is true of the magnesia salts.

#### XIV. MANGANESE.

*Manganous chloride*,  $2C_6H_7N.MnCl_2$ .—An alcoholic solution of two molecules of aniline was added to one molecule of manganous chloride likewise dissolved in alcohol. A cloudiness formed immediately, and the compound slowly crystallized out as beautiful white rhombic plates, with a pearly luster.

When the compound with manganous chloride was formed by direct addition of aniline, the temperature rose from  $18^{\circ}$  to  $24^{\circ}$ .

*Manganous bromide*,  $2C_6H_7N.MnBr_2$ .—By direct mixing of the constituents, a rise of temperature from  $21^{\circ}$  to  $40^{\circ}$  resulted. It separated from alcohol in small colorless crystals.

*Manganous iodide*,  $2C_6H_7N.MnI_2$ .—The temperature rose from  $21^{\circ}$  to  $39^{\circ}$ . The small, colorless crystals obtained from the alcoholic solution were similar to the bromine compound.

*Manganous sulphate.*—It did not enter into combination.

#### XV. MERCURY.

*Mercuric chloride*,  $2C_6H_7N.HgCl_2$ .—"It is precipitated on adding mercuric chloride to an excess of alcoholic aniline. It is a nacreous precipitate, which must be collected on a filter and washed with a small quantity of alcohol. It gives off a small quantity of aniline

even at  $60^{\circ}$ , and turns yellowish (Gerhardt, *Traité*, 3, 86). According to Schiff, it decomposes at  $100^{\circ}$ , yielding aniline-red, and a similar red product is obtained by heating aniline with mercurous chloride to  $150^{\circ}$ . (Probably the aniline used contained toluidine.)" Watts' Dict., 4, 475.

I obtained the salt by heating the aniline directly with mercuric chloride and crystallizing out of ammonium chloride. The crystals were several millimeters in length, and of a yellowish-white color. They were not pure, and the compound is best obtained by heating aniline directly with mercuric chloride. It crystallized from alcohol in small white needles. The combination with mercuric chloride caused an elevation of temperature from  $24^{\circ}$  to  $106^{\circ}$ .

$\text{NC}_6\text{H}_5\text{.Hg.HCl}$ .—According to Foster (*Ber. der Deutsch. Chem. Gesell.*, 7, 294), this compound is produced together with  $2\text{C}_6\text{H}_7\text{N.HgCl}_2$  when the boiling solutions of aniline and mercuric chloride are mixed together. The  $2\text{C}_6\text{H}_7\text{N.HgCl}_2$  remains in solution, whilst the new compound, a "phenylated white precipitate," settles as a yellow precipitate, and by repeated exhaustion with boiling alcohol may be obtained pure. The above formulæ was in accordance with the analysis :—

	<i>Theory.</i>	<i>Found.</i>
C .....	21.98	21.60
H.....	1.83	2.00
N.....	4.28	4.52
Cl.....	10.84	11.15
Hg.....	61.07	60.96-61.47.

*Mercurous chloride*.—It could not be made to enter into combination.

*Mercuric iodide*,  $2\text{C}_6\text{H}_7\text{N.HgI}_2$ .—After dissolving mercuric iodide in boiling aniline, the solid mass which formed on cooling was dissolved in boiling ammonium chloride, filtered and crystallized. Prismatic needles were formed of considerable size, which decomposed on attempting to further purify them by recrystallization from water.

According to Schiff, this compound forms little yellowish leaves, which can be obtained from the nitrate by double decomposition.

This statement of Schiff could not be verified. Neither could a compound be crystallized out from a solution of the mass, formed by heating aniline with mercuric iodide, in alcohol. No evidence of the existence of a compound of these two bodies could be obtained.

Klein states that he formed the compound  $2\text{C}_6\text{H}_7\text{N}.\text{HgI}_2$  with a melting point of  $60^\circ$ .

*Mercuric bromide*,  $2\text{C}_6\text{H}_7\text{N}.\text{HgBr}_2$ .—No attempt to make it. Formed, according to Klein, in long crystals when crystallized out of alcohol. M. pt.,  $110^\circ$ – $112^\circ$ . Decomposed by boiling water.

*Mercuric nitrate*,  $2\text{C}_6\text{H}_7\text{N}.\text{Hg}(\text{NO}_3)_2$ .—According to Schiff, this is obtained as a white precipitate, when aniline is added to a solution of mercuric nitrate. The powder becomes crystalline if it is digested, while still moist, with cold dilute nitric acid. Small brilliant leaves are obtained, if a solution of the slightly acidulated nitrate is used, or if the neutral mercuric solution is added drop by drop to an acidulated solution of nitrate of aniline. In every case the same compound is formed and not the double salt. The salt is unalterable in the air, and slightly soluble in dilute nitric acid.

“When heated with water, the salt yields aniline nitrate and changes to a dense white crystalline powder,  $\text{C}_6\text{H}_5.\text{NH}.\text{HgNO}_3$ .”

“This last salt, diffused during several days with a large quantity of boiling water, gives up more aniline nitrate, and forms  $(\text{C}_6\text{H}_5\text{N})_2.\text{Hg}_2(\text{NO}_3)_2 + 2\text{H}_2\text{O}$ . When heated with water for eight more days, this last compound suffers no further alteration.”

*Mercurous nitrate*.—“Mercurous nitrate likewise combines with aniline to form  $2\text{C}_6\text{H}_7\text{N}.\text{Hg}_2(\text{NO}_3)_2$ , a white crystalline powder, very readily decomposed by a slight elevation of temperature, with reduction of the mercury.”

The above statements of Schiff, with regard to the compounds of mercuric and mercurous nitrate, could not be verified, and no proof was obtained of the existence of any of the bodies above spoken of.

When mixed with aniline, the mercurous nitrate caused an elevation of temperature from  $21^\circ.5$  to  $79^\circ.5$ , and the mass increased greatly in volume, forming a black crystalline substance. After removing the excess of aniline it was dissolved in alcohol, from which it did not crystallize satisfactorily.

This fact, together with the circumstance that the crystals in aqueous solution gave no reaction for aniline, shows that no compound was formed.

*Mercuric cyanide*,  $2\text{C}_6\text{H}_7\text{N}.\text{HgCy}_2$ .—On adding powdered mercuric cyanide to aniline, the rise of temperature was only from  $17^\circ$  to  $21^\circ$ , and in order to insure complete combination, the mixture was heated to the boiling point of the aniline. The latter being in excess, it dissolved the resulting compound at the same time that it

entered into combination with the cyanide, and on cooling and standing the  $2C_6H_7N.HgCy_2$  was deposited as stout prisms, six-sided, and of a vitreous luster. These crystals were freed from adhering aniline by washing with alcohol.

According to Schiff,  $2C_6H_7N.HgCy_2$  separates in long, magnificent needles on mixing aniline with a hot aqueous solution of mercuric cyanide. It melts very readily, and is resolved into aniline and cyanide of mercury at  $80^\circ$ . It is attacked neither by alkalies nor by biniodide of potassium.

#### XVI. NICKEL.

*Nickel chloride*,  $2C_6H_7N.NiCl_2$ .—It united directly with aniline, the temperature rising from  $20^\circ$  to  $40^\circ$ . The product solidified to a dark green mass, which crystallized out of alcohol in clusters of green needles. It altered in color on keeping.

According to Lippmann and Vortmann, who prepared this compound somewhat differently, the body obtained from alcohol contains  $2C_6H_7N.NiCl_2.2C_6H_6O$ , and gives off alcohol at  $100^\circ$ , becoming yellowish-green, and forming  $2C_6H_7N.NiCl_2$ .

*Nickel sulphate*.—Does not enter into combination.

#### XVII. PALLADIUM.

Palladium chloride formed a compound, but the amount of the salt at my disposal was too small to admit of study.

#### XVIII. PLATINIC CHLORIDE AND PLATINOUS CHLORIDE.

The former does not combine. The compounds of the latter will be found described in Beilstein's Handb. der Organ. Chemie, p. 875.

#### XIX. POTASSIUM.

No combination ensued between aniline and potassium iodide, or with the other salts of potassium experimented upon.

#### XX. STRONTIUM.

Its salts do not combine with aniline.

#### XXI. TIN.

*Stannous chloride*,  $2C_6H_7N.SnCl_2$ .—"It is obtained by mixing the same number of equivalents of stannous chloride and aniline. A slight evolution of heat occurs, and at the end of some hours a crystalline mass is obtained which is slightly soluble in cold water and in alcohol, but which is decomposed by these liquids on warming" (Schiff).

On mixing aniline with stannous chloride the temperature rose from  $22^{\circ}$  to  $35^{\circ}$ . The compound crystallized out of alcohol in small white needles (Leeds).

*Stannic chloride*,  $4\text{C}_6\text{H}_7\text{N}.\text{SnCl}_4$ .—"It has been obtained by the union of 2 equivalents of aniline to 1 eq. of stannic chloride. An intense heat is produced at the moment of mixture, and it is necessary, to avoid explosions, to place the vase in a cooling mixture. Under these conditions, a perfectly white crystalline mass is obtained" (Schiff).

"Or if one adds the aniline drop by drop to a solution of stannic chloride in benzine, a white crystalline powder is seen to deposit at once, which is the  $4\text{C}_6\text{H}_7\text{N}.\text{SnCl}_4$ , insoluble in benzine. It is decomposed by water, stannic hydrate being precipitated" (Schiff).

When stannic chloride was added, the temperature rose from  $21^{\circ}$  to  $60^{\circ}$ . Its crystallization from alcohol yielded small white needles, turning bluish-gray after a long interval (Leeds).

*Stannous iodide*,  $2\text{C}_6\text{H}_7\text{N}.\text{SnI}_2$ .—The temperature rose from  $21^{\circ}$  to  $40^{\circ}$ , and the compound crystallized out of alcohol in grayish-white needles.

## XXII. TITANIUM.

*Titanium chloride*,  $4\text{C}_6\text{H}_7\text{N}.\text{TiCl}_4$ .—The flask which contained the titanium chloride was broken, and the vapor allowed to flow down in contact with the aniline. It was energetically absorbed, forming a hard mass of a reddish color. This was dissolved in alcohol, the compound separating out in white, feathery crystals.

## XXIII. URANIUM.

*Uranium acetate*.—Formed no compound.

*Uranyl chloride*,  $2\text{C}_6\text{H}_7\text{N}.\text{UO}_2\text{Cl}_2$ .—The chloride in alcohol solution was added to the alcoholic solution of aniline, and allowed to stand. The compound was slowly precipitated out as small yellow needles. Owing to their solubility in alcohol they were washed with ether, and afterwards recrystallized from alcohol. No elevation of temperature was noted on mixing the alcoholic solutions.

Uranium nitrate entered into combination under the same circumstances as the chloride, but the precipitate formed immediately on mixing the alcoholic solutions, as a heavy yellow crystalline compound.

## XXIV. ZINC.

*Zinc chloride*,  $2\text{C}_6\text{H}_7\text{N}.\text{ZnCl}_2$ .—"Crystallizes in oblique, rhomboidal prisms, anhydrous, soluble in water and in alcohol, especially in

the cold. By prolonged boiling of these solutions the salt is decomposed into aniline and zinc chloride. It combines with platinic chloride, forming granular crystals" (Schiff).

*Zinc iodide*,  $2C_6H_7N.ZnI_2$ .—On adding the zinc salt to an excess of aniline, the temperature rose from  $17^\circ.5$  to  $63^\circ$ , and the substances united to form a white mass, which became very hard and compact. The excess of aniline was removed by washing with a small amount of alcohol. The residue was dissolved by digesting with alcohol under a return cooler, and after two recrystallizations became quite pure. It formed unusually brilliant prismatic needles of about a centimeter in length, with a pearly luster, and aggregating into radiate clusters.

*Zinc bromide*,  $2C_6H_7N.ZnBr_2$ .—It was prepared in like manner, the temperature of the mixture rising from  $16^\circ$  to  $62^\circ$ . The crystals were similar to those of the foregoing compound, but not so long or brilliant.

*Zinc sulphate*.—"It is more soluble than the preceding salts, and can be used with advantage to prepare them by the method of double decomposition" (Schiff).

I did not succeed in forming a compound with zinc sulphate.

### *Compounds with Paratoluidine.*

#### I. CADMIUM.

*Cadmium nitrate*,  $2p-C_7H_9N.Cd(NO_3)_2$ .—The first method employed in making this compound was by the addition of an aqueous solution of paratoluidine nitrate to an aqueous solution of cadmium nitrate, both salts being in molecular proportions. But the toluidine nitrate crystallized out separately.

The second method was by heating the cadmium nitrate directly with paratoluidine. A solid white mass was formed, which yielded, when recrystallized from benzine or alcohol, white needles compacted together like leaves.

*Cadmium bromide*,  $2p-C_7H_9N.CdBr_2$ .—It was prepared by mixing together the alcoholic solutions of the two bodies in molecular proportions, and precipitated as a dense white crystalline mass.

*Cadmium iodide*,  $2p-C_7H_9N.CdI_2$ .—From the alcoholic solutions it was precipitated in white prismatic needles, which were washed with alcohol. It was also formed by heating the cadmium iodide directly with fused paratoluidine, and recrystallization from alcohol. The temperature of the fused mass rose from  $67^\circ$  to  $90^\circ$ .

## II. COPPER.

On the addition of paratoluidine nitrate in aqueous solution to an aqueous solution of cupric nitrate, a dark green precipitate was obtained, which speedily blackened. On standing, crystals of impure toluidine nitrate formed on the sides of the vessel, and a blackish mass containing copper and some organic matter was left behind. But no definite compound could be isolated.

A similar change took place in the compound formed on the addition of cupric chloride to paratoluidine hydrochlorate, an uncrySTALLINE blackish mass remaining.

## III. COBALT.

On mixing the aqueous solutions of the nitrates, no compound was formed, the constituents crystallizing out separately, with the formation of a small amount of a blackish mass, due to partial decomposition.

The chlorides, likewise, did not enter into combination when their aqueous solutions were mixed together.

Lippmann and Vortmann have already explained the reaction\* when an alcoholic solution of cobaltous chloride is added to paratoluidine.

## IV. CHROMIUM.

*Chromium sulphate*.—After mixing the aqueous solutions of the two salts, they were allowed to evaporate spontaneously, when the constituents crystallized out separately.

*Chromic chlorides*.—A solution of chromic acid was reduced by alcohol and hydrochloric acid to  $\text{Cr}_2\text{Cl}_6$ , and added to an alcoholic solution of paratoluidine. The solutions solidified to a brown mass, from which, on treatment with water, the toluidine chloride crystallized out, leaving the chromium sesquichloride behind as a green powder.

## V. GOLD.

One mol. of auric chloride dissolved in alcohol, was added to three molecules of toluidine dissolved in the same solvent. The liquid became dark purple and after a time deposited a sediment due to the reduction of the gold salt.

## VI. MERCURY.

*Mercurous nitrate*,  $2p\text{-C}_7\text{H}_7\text{N.Hg}_2(\text{NO}_3)_2$ .—It was formed on adding an aqueous solution of toluidine nitrate to an aqueous solution

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\* *Ber. der Deutsch. Chem. Gesell.*, 12, 81.



of mercurous nitrate, acidified with nitric acid. The salt separated out slowly in small opaque white needles. It was purified by washing with cold water; in hot water, acidified with nitric acid, it dissolved readily.

*Mercuric nitrate*.—The corresponding mercuric salt could not be obtained in a crystalline condition. The precipitate obtained on evaporating the aqueous solutions of toluidine nitrate and mercuric nitrate to a small bulk, was easily soluble in water, but would not crystallize from the solution.

*Mercuric chloride*,  $2p\text{-C}_7\text{H}_7\text{N.HgCl}_2$ .—It was obtained by adding 2 mols. of toluidine dissolved in alcohol to 1 mol. of mercuric chloride, likewise in alcoholic solution, and dissolving the precipitate in boiling ammonium chloride. It crystallized out in white needles an inch in length, which decomposed on treatment with water. For this reason the toluidine and mercuric chloride were heated together until combination ensued, the resulting compound dissolved in alcohol, and recrystallized from the same solvent. The crystals in this case were white also, but not of such great length as those obtained from the ammonium chloride solution.

According to Klein, it crystallizes out of ether in magnificent thick needles, and decomposes at  $70^\circ$ , giving off paratoluidine.

*Mercuric cyanide*,  $2p\text{-C}_7\text{H}_7\text{N.HgCy}_2$ .—Formed like the corresponding aniline compound, and crystallizing similarly. The crystals are much larger and have an adamantine luster.

#### VII. MANGANESE.

$2p\text{-C}_7\text{H}_7\text{N.MnCl}_2$ .—This was prepared both by adding the alcoholic solutions of the two components, and by adding the manganese chloride to fused paratoluidine. It could not be obtained in pure white crystals, but only as a faint red crystalline powder.

#### VIII. SILVER.

One mol. of argentic nitrate dissolved in water was added to an aqueous solution of toluidine nitrate. No combination occurred, the toluidine nitrate crystallizing out in long white needles.

#### IX. URANIUM.

$2p\text{-C}_7\text{H}_7\text{N.UO}_2\text{Cl}_2$ .—The attempt to prepare the salt by mixing the aqueous solution of uranyl chloride with an aqueous solution of toluidine hydrochlorate was unsuccessful: the yellow solution deposited a pink powder, while the toluidine hydrochlorate crystallized out.

No better result was obtained with the alcoholic solutions of uranyl chloride and of toluidine. The uranium salt separated out on standing as a yellow powder, and the toluidine alone crystallized from the alcohol.

In a third trial the alcoholic solutions of the two bodies in molecular proportions were mixed together, the alcohol evaporated off, and the residue dissolved in aniline. After long standing the compound crystallized out in long, thick, greenish-yellow prisms, apparently orthorhombic and doubly terminated. They were drained from the excess of aniline by pressing with filter paper, and washed with a little alcohol.

#### X. ZINC.

*Zinc bromide*,  $2p\text{-C}_7\text{H}_9\text{N}\cdot\text{ZnBr}_2$ .—Formed by adding to two molecules of fused paratoluidine, one molecule of zinc bromide. The temperature rose during the formation of the compound from  $57^\circ$  to  $89^\circ$ . By crystallization from alcohol a large yield of delicate white needles was obtained.

*Zinc iodide*,  $2p\text{-C}_7\text{H}_9\text{N}\cdot\text{ZnI}_2$ .—This was formed in the same manner as the preceding, the temperature of the fused mass rising from  $61^\circ$  to  $91^\circ$ . It crystallized from alcohol in long, extremely slender white needles, having a satin luster, and forming fan-like clusters.

For other compounds, see Beilstein's Lehrbuch, p. 936.

#### *Compounds with Orthotoluidine.*

##### I. CADMIUM.

$2o\text{-C}_7\text{H}_9\text{N}\cdot\text{CdI}_2$ .—Formed by adding the cadmium iodide directly to the orthotoluidine. The temperature rose from  $18^\circ$  to  $45^\circ$ . It crystallized from alcohol in fan-like aggregates of soft, small needles.

##### II. MERCURY.

*Mercuric chloride*,  $2o\text{-C}_7\text{H}_9\text{N}\cdot\text{HgCl}_2$ .—The temperature at moment of formation rose from  $20^\circ$  to  $49^\circ.5$ . It crystallized from alcohol in leaf-like crystals, having very much the appearance and luster of small tufts of mica.

*Mercuric cyanide*,  $2o\text{-C}_7\text{H}_9\text{N}\cdot\text{HgCy}_2$ .—Formed similarly. Separated in beautiful crystals, with superb adamantine luster. The temperature at the moment of formation rose from  $18^\circ$  to  $21^\circ$ .

##### III. MANGANESE.

$2o\text{-C}_7\text{H}_9\text{N}\cdot\text{MnCl}_2$ .—Formation-temperature rose from  $18^\circ$  to  $22^\circ$ . The compound was with difficulty obtained in a pure condition from the alcoholic solution, the crystals resembling the corresponding paratoluidine compound.

## IV. ZINC.

*Zinc bromide*,  $2o\text{-C}_7\text{H}_9\text{N.ZnBr}_2$ .—The temperature of the mixed components rose from  $18^\circ$  to  $59^\circ$ , with increase of volume. Formed compact white crystalline masses.

*Zinc iodide*,  $2o\text{-C}_7\text{H}_9\text{N.ZnI}_2$ .—The temperature rose from  $17^\circ$  to  $58^\circ$ . Tufts of fine white needles.

*Compounds with Metaxylidine.*

## I. CADMIUM.

*Cadmium bromide*,  $2\text{C}_8\text{H}_{11}\text{N.CdBr}_2$ .—Formed in the same manner as the corresponding aniline and toluidine compounds. The temperature rose from  $17^\circ$  to  $42^\circ$ . A pinkish crystalline powder. The color was probably due to alteration in contact with the air. As first crystallized from alcohol, it was in small white needles.

*Cadmium iodide*,  $2\text{C}_8\text{H}_{11}\text{N.CdCl}_2$ .—Formation-temperature rose from  $18^\circ$  to  $42^\circ$ . Separated from alcohol in minute white crystals.

## II. MERCURY.

*Mercuric chloride*,  $2\text{C}_8\text{H}_{11}\text{N.HgCl}_2$ .—The temperature rose from  $18^\circ$  to  $71^\circ$ . It crystallizes from alcohol in white needles.

*Mercuric cyanide*,  $2\text{C}_8\text{H}_{11}\text{N.HgCy}_2$ .—Minute crystals, with superb adamantine luster, and with faces apparently of a rhombohedral character.

## III. ZINC.

*Zinc bromide*,  $2\text{C}_8\text{H}_{11}\text{N.ZnBr}_2$ .—The temperature rose from  $17^\circ.5$  to  $61^\circ.5$ , with a great increase of volume. Crystallizes from alcohol in very small needles, which compact themselves together in white botryoidal masses.

*Zinc iodide*,  $2\text{C}_8\text{H}_{11}\text{N.ZnI}_2$ .—The temperature rose from  $16^\circ$  to  $59^\circ$ . Crystallizes from alcohol in small white needles, aggregated into hemispherical rosettes.

*Compound with Dimethylaniline.*

*Mercuric chloride*,  $2\text{C}_6\text{H}_5(\text{CH}_3)_2\text{N.HgCl}_2$ .—One molecule of mercuric chloride was added to two molecules of dimethyl-aniline, the temperature of the mixture rising from  $21^\circ$  to  $44^\circ$ . The solid mass formed on cooling was thoroughly washed with alcohol and was left as a greenish-gray powder.

*Compound with Naphthylamine.*

*Mercuric chloride*,  $2\text{C}_{10}\text{H}_9\text{N.HgCl}_2$ .—After fusion of the naphthylamine, and addition of the mercuric chloride, the temperature of

the mixture rose from  $61^{\circ}$  to  $67^{\circ}$ . The resulting mass was washed repeatedly with alcohol without becoming white, but it crystallized from boiling ligroine in minute white needles, turning brown in contact with the air.

*Note on Thiocarbanilide.*

It was found that the compound  $2C_6H_7N.HgCl_2$  underwent rapid decomposition when exposed in contact with carbon disulphide to the action of sunlight. Subsequently, that this decomposition took place merely by heating, and could be best effected in the following manner :

The purified chloride of diphenylmercurammonium was dissolved in alcohol, and after being mixed with carbon disulphide, was boiled for several days under a return cooler. When the reaction was finished, which was made evident by the clearing of the liquid, the precipitated  $HgS$  was filtered off, and the filtrate set aside to crystallize.

After a second crystallization, a pure product was obtained with a melting point of  $144^{\circ}$ , and which proved on analysis to be thiocarbanilide,  $C_{13}H_{12}N_2S$ .

	<i>Found.</i>	<i>Theory.</i>
Carbon.....	68.31	68.42
Hydrogen.....	5.41	5.26
Sulphur.....	14.00	14.04

Thiocarbotoluidide and thiocarboxylidide were formed in the same manner from  $2C_7H_9N.HgCl_2$  and  $2C_8H_{11}N.HgCl_2$ .

The cyanide of diphenylmercurammonium also yields thiocarbanilide when boiled with carbon disulphide, hydrocyanic acid escaping.

Thiocarbanilide is ordinarily prepared by boiling aniline and alcoholic potash with an excess of carbon disulphide.

### XXXV.—ANALYSIS OF BEET ROOT AND SORGHUM CANE.

BY P. CASAMAJOR.

In the United States, for the last twenty years, many attempts have been made to manufacture sugar in a commercial way, from beet root and from the sorghum cane. Such attempts continue to be made at this day, and although there is no predicting what results

may be obtained in the future, I believe that heretofore these attempts have not been successful.

There are many persons who believe that cane sugar can be made commercially from the sorghum cane, while others believe that beet root is preferable to sorghum. The partisans of beet and those of sorghum differ widely in their estimates of the comparative values of these two sources of sugar. There is only one way of settling the question, which is to produce beet or sorghum which will yield good results. I have had occasion to examine several samples of beet root raised in the Northern States, and one of sorghum, and the results obtained were in all cases very unfavorable.

The object of this communication is to call attention to the processes by which we may ascertain beforehand the quantity of sugar which may be obtained from a given weight of beet root or of sorghum cane. It is nearly useless to try to ascertain the yield of a saccharine juice by experiments on a small scale, as such experiments never yield results analogous to those which can be obtained on a large scale with improved apparatus.

Beet root or sorghum cane can only be considered by the sugar manufacturer as so much raw material. To estimate the value of either, the manufacturer should follow exactly the course that a refiner follows to ascertain the commercial value of a raw sugar. A refiner knows that, from a given quantity of raw sugar, he can obtain a certain portion of pure or nearly pure sugar, and a certain quantity of molasses which will not yield any crystallizable sugar. Therefore, if he can ascertain the total quantity of sugar in raw sugar, and the portion which will remain in the molasses, the difference will be the pure sugar which he will obtain in refining.

It may be urged, that account should also be taken of a certain quantity of sugar lost during the process of refining, but this loss is due to causes entirely independent of the composition of any raw sugar under examination, and it should be left out of the calculation.

The composition of molasses from a well regulated sugar-house is pretty nearly constant. If raw cane sugars are used in a refinery, the composition of the molasses which will not yield any sugar crystals will be very nearly as follows for a syrup of density equal to 40° Baumé :

Sugar.....	37.5
Soluble impurities.....	37.5
Water.....	25.0

A refiner may obtain, as residue, a syrup containing a smaller percentage of sugar, but we may assume, for the purpose of comparing one sample of raw sugar with another, that the above will be the composition of the molasses obtained in refining.

For the sake of convenience in comparing one product with another in a sugar refinery, the composition of each product is sometimes stated *in the dry state*, or on the supposition that the product contains no water. Water is easily added, and it may be taken away by evaporation without affecting the quantity of pure sugar obtainable from a given commercial sugar. The quantity of sugar in a product reduced to dryness, compared to the total substances, is called *coefficient of purity* or *quotient of purity*.

Referring to the composition of the sugar-house molasses given above, we may see that its *coefficient of purity* is 50, which means that in cane sugars 1 per cent. of soluble impurities prevents the crystallization of 1 per cent. of sugar.

It has been argued that the coefficient of purity of molasses is not a safe guide for calculating the yield of pure sugar from a given sample of raw material.

There would be some foundation for this if the coefficient of purity of the molasses of a given refinery varied within very wide limits. The contrary is, however, the case. Thousands of tests, extending through several years, have convinced me that the coefficient of purity of molasses may be kept within very narrow limits. The coefficient is not always 50. Some refiners prefer to keep it somewhat lower; but the coefficient 50 answers very well for calculating the quantity of pure sugar obtainable from a raw cane sugar.

To apply the above, let us suppose that we have a raw sugar whose coefficient of purity is 92. The composition of the sugar, supposed dry, would be :

Sugar.....	92
Impurities.....	8

As 8 parts of impurities prevent the crystallization of 8 parts of sugar, the yield of the above dry sugar would be  $92 - 8 = 84$  per cent.

The results which can be obtained from a given weight of beet root sugar are somewhat different from those which cane sugar will afford. A series of tests made in the principal sugar refineries of Paris show that the coefficient of purity of beet molasses seldom goes below 52, and is generally 55. From this we may deduce the rule that in beet root sugar 1 per cent. of impurities prevent the crystallization of 1.2 per cent. of sugar.

The composition of sorghum molasses, from which all the crystallizable sugar has been separated, has not been ascertained, as far as I know. From the analogy between sorghum and the sugar cane we may assume that, for sorghum sugar, 1 per cent. of soluble impurities prevents the crystallization of 1 per cent. of sugar.

The difficulty with beet root and sorghum cane grown in this country is principally that the coefficient of purity of their juices is not sufficiently high. Whenever a product is obtained whose coefficient of purity is high enough, the quantity of juice in a certain weight of roots or of cane may be ascertained, and this presents no difficulty. If, however, the coefficient of purity is not sufficiently high, there is no use in making further determinations, as there cannot be any profit in working a juice of low grade.

The coefficient of purity of a juice is determined very quickly and accurately by using a process which I first described in the *American Chemist* for October and November, 1873. This process is described more fully and in better form in the *Moniteur Scientifique* for March, 1877. In a late book on sugar analysis, published by Van Nostrand, it is said that this process is advisable when a balance is not at hand, but that it does not give as satisfactory results as Balling's process, which is mentioned as the *direct* process.

As my process is in use in most of the important refineries in the United States, I do not feel called upon to defend it. A constant use of it for several years shows that the results it gives agree within at least 1 per cent., and generally within less than  $\frac{1}{2}$  per cent. This process is founded on this: That the pure sugar in 100 c.c. of a solution is given by multiplying the indication of the optical saccharometer by 0.26048 when a Ventzke instrument is used, or by 0.1635 for the Duboscq saccharometer. On the other hand, the total quantity of substance in solution is represented by the degree Balling, multiplied by the specific gravity.

The coefficient of purity is obtained by dividing the first product by the second. If we call the saccharimetric test S, the specific gravity P, and the Balling degree B, we shall have—

$$\text{Coefficient of purity} = \frac{S \times 0.26048}{B \times P}$$

This may be written :

$$S \times \frac{0.26048}{B \times P}$$

If for every degree Balling we calculate the quantity

$$\frac{0.26048}{B \times P}$$

we may form a table of factors by which to multiply the saccharimetric degree to obtain the coefficient of purity of a solution.

We may, instead of the Balling degree, determine the specific gravity, and for any specific gravity use the factor corresponding to

$$\frac{0.26048}{B \times P}$$

In the articles above referred to, tables are given for Balling degrees between 5° and 15°, and for Geisler's specific gravity spindle, ranging from 1.095 to 1.1050. For any other degree Balling, or any other specific gravity, the factors may be easily calculated from the formula given above.

The specific gravity may be determined by a balance, and the factor may be obtained by the following formula, in which the Balling degree does not enter :

$$\frac{0.1}{P - 1}$$

in which P is the specific gravity. This is for Ventzke's instrument For the Duboscq saccharometer, the factor is given by

$$\frac{0.0628}{P - 1}$$

To test a lot of beets for coefficient of purity, the juice should be taken so as to represent the average juice of the lot. A wedge-shaped piece should be cut out of every root picked out for analysis. The thin edge of the wedge should be at the axis of the root, and the angle of each wedge should be the same for every piece. All the wedges may be rasped on a common grater, and the pulp obtained should be pressed in a cloth so as to obtain the juice. This is immediately neutralized with lime, and heated, by which a thick black precipitate is obtained. The liquid, after filtration, is light and clear enough to be placed in the tube of the optical saccharometer. The density of the filtered liquid should be taken to determine the coefficient of purity, as the juice of beets can always be defecated with a little lime.

The juice of sorghum cane is easily obtained by crushing with a hammer and wringing the cane with the hands, if no special apparatus is at hand.



If the coefficient of purity of a juice is sufficiently high, we may in the next place determine the quantity of juice in a certain weight of the raw material.

The quantity of juice in the pulp obtained by rasping may be got at by drying a certain weight of the pulp and determining the water in the pulp by loss. We also determine the water in the juice by taking the difference between 100 and the Balling degree. As all the water in the pulp is in the juice, we have :

$$\text{per cent. juice in the pulp} = \frac{\text{per cent. of water in the pulp.}}{\text{per cent. of water in the juice.}}$$

As to what constitutes a sufficiently high coefficient of purity, this should be left to the judgment of the parties interested. I may, however, call to attention that in the North of France in 1879, a beet was not considered as fit to work for sugar, the coefficient of purity of whose juice was below 79. For 100 parts of dried juice the composition would be :

Pure sugar.....	79
Impurities.....	21

We have seen that in beet juice 1 part of impurities prevents the crystallization of 1.2 parts of sugar, therefore the yield would be :

$$79 - (21 \times 1.2) = 79 - 25.2 = 53.8 \text{ per cent.}$$

I have had occasion to analyze several samples of beet roots grown in the United States. Those which gave the highest coefficient of purity were from Delaware, the coefficient of purity of the juice being 63 per cent. The yield of sugar of the dried juice would be

$$63 - (37 \times 1.2) = 63 - 44.4 = 18.6 \text{ per cent.}$$

Most of the beet roots I tested had a coefficient of purity below 50, and they could not, therefore, yield any sugar.

The coefficient of purity of the juice of a sorghum cane which I tested lately was 44, and of course no sugar could be obtained from this juice.

It is almost unnecessary to state in conclusion, that the processes described are applicable to the sugar cane, and to other plants with saccharine juices.

## Reports on American and Foreign Patents Relating to Chemistry.

### American Patents.

Condensed from the Official Gazette of the U. S. Patent Office, by ARNO BEHR.

*May 3, 1881.*

240,846.—*Process of melting and purifying glass.* JACOB REESE.

240,855.—*Manufacture of glue.* FRIEDRICH SELTSAM and RICHARD HAGEN.

240,879.—*Process of purifying saccharine liquids of low grades.* ARNO BEHR, OTTO H. KRAUSE, and HENRY E. NIESE.

The solutions diluted to about 15° Bé are treated with lime and slightly heated, the flocculent precipitate is removed by filtration, and the caustic lime contained in the filtrate saturated with carbonic acid, and the liquid heated and filtered. The first and second precipitates contain considerable amounts of coloring and other organic matters.

240,936.—*Apparatus for, and process of, cooling and refining oil.* WM. G. WARDEN.

Air which has been dried with sulphuric acid, and cooled, is driven through the oil.

240,941 and 240,942.—*Manufacture of artificial indigo.* ADOLPH BAYER.

Two methods for the artificial production of indigo, the first from isatine chloride with glacial acetic acid and zinc dust, the second from isatine with phosphortrichloride, acetylchloride, and yellow phosphorus.

241,056.—*Manufacturing artificial wool from vegetable fiber.* MICHAEL NEUMANN.

241,070.—*Photographic paper.* THOMAS C. ROCHE.

*May 10, 1881.*

241,163.—*Blasting powder.* TRUE P. SLEEPER.

Chlorate of potash, sugar, and charcoal.

241,164. *Process of smelting ores containing iron and precious metals.* HENRY A. SPEARS.

Mixing the ores with lead or lead-ores, and separately tapping off the pig-iron and base bullion.

241,172.—*Centrifugal machine for creaming milk.* DAVID M. WESTON.

241,202.—*Manufacture of glucose.* AMBROSE G. FELL.

The inventor claims the use of nitric acid together with sulphuric acid for conversion; conversion at a temperature below the boiling point of water; use of sulphurous acid gas in the vacuum pan.

241,203.—*Filtering tank.* AMBROSE G. FELL.

241,207.—*Treating sorghum or ribbon cane.* WILLIAM L. GEBBY.

The cane is scalded with steam and then dried.

241,216.—*Process of fusing and moulding iridium.* JNO. HOLLAND.

“Raising the metal to a high heat, then adding to it phosphorus, and after the metal is cast releasing the phosphorus by subjecting the metal to heat in a chalk bath.”

241,383.—*Perfumed caustic soda.* GEORGE T. LEWIS.

Add from 10 to 30 drops of an essential oil to every pound of granulated or pulverized caustic soda.

241,398.—*Mordant.* SAMUEL MELLOR.

“Stibio-fluorine salts.”

241,463.—*Manufacture of fertilizers from blood.* RICHARD WERDERMANN.

“Adding lime to the fresh blood, agitating the mixture, next precipitating the lime, and finally drying the coagulated blood.”

*May 17, 1881.*

241,476.—*Treating cereals to separate the oily germs, flour, and starch for the use of distilleries, etc.* FERDINAND CAMUS.

Maize is soaked in a solution of a sulphite or bisulphite, and afterwards treated with a dilute mineral acid. To the sulphurous acid liberated in this way within the grain and which is called nascent sulphurous acid, the inventor ascribes a peculiar action on the grain, which facilitates a subsequent separation of the germs and hulls from the mealy part of the grain.

241,505.—*Jellifying petroleum and other substances insoluble in water.* CYPRIEN TESSIÈ DU MOTAY, AUGUSTE J. ROSSI, ALEXANDRE BOURGOUGNON, and PAUL CASAMAJOR.

The process consists in adding petroleum gradually, while agitating, to a solution of gelatine.

241,570.—*Process of lining Bessemer converters or other furnaces, and the preparation of lime used therefor.* SIDNEY G. THOMAS and PERCY C. GILCHRIST.

241,655.—*Lactometer.* FRIEDRICH HEEREN.

The color of a thin layer of milk viewed against a black background is compared with a scale of colors conveniently arranged.

241,661.—*Dyeing colors on cotton or textile fabrics.* THOMAS and ROBERT HOLLIDAY.

Azocolors produced by the action of phenoles upon diazo compounds are developed directly upon the cloth by impregnating it with the two solutions in succession.

- 241,677.—*Process of preserving organic substances.* CHARLES F., ARTHUR W., and ALBERT L. LAWTON.

Fruits are placed in large receptacles, which are then filled with a mixture of nitrogen and carbonic oxide.

- 241,713.—*Method of preparing paper for reproducing drawings and designs.* HENRI PELLET.

The paper is impregnated with a mixture of a ferric salt and citric acid, and covered with the drawing to be reproduced, exposed to the action of light. Subsequent washing with a solution of yellow prussiate of potash develops the design in blue lines.

- 241,718.—*Process of manufacturing solid steel castings.* JACOB REESE.

- 241,738.—*Manufacture of artificial chinoline.* ZDENKO H. SKRAUP.

Chinoline is produced by heating a mixture of nitrobenzole, aniline, glycerine, and sulphuric acid.

- 241,772.—*Process for the treatment of flax-seed.* HOMER T. YARYAN.

Moistens the seed before it is extracted, with naphtha.

*May 24, 1881.*

- 241,810.—*Composition of matter for the manufacture of illuminating candles.* RICHARD F. W. LOPER.

Wax, carbon oil, and animal charcoal.

- 241,868.—*Treating phosphates for fertilizers.* GUSTAV A. LIEBIG.

"Treating phosphates by mixing them with carbon and subjecting them to heat, for fertilizing purposes."

- 241,897.—*Manufacture of size.* EMIL R. VON PORTHEIM.

Coagulated or insoluble albuminoid substances are dissolved in an alkali, and the excess of the latter is neutralized with muriatic acid.

- 241,903.—*Process of manufacturing crystallizable grape sugar.* HORACE WILLIAMS and JOHN L. ALBERGER.

Corn is treated with hot dilute acid, and the resulting saccharine liquid is treated in the usual way until the final concentration, after which the inventors claim to have added the following steps, viz.: cooling of the concentrated syrup, adding a small percentage of powdered grape-sugar, and pressing the mass after crystallization.

- 241,941.—*Method of preparing nitro-glycerine compounds.* GILBERT S. DEAN.

100 pts. nitro-glycerine, 10 pts. of pulverulent nitro-cellulose, and 2 to 3 pts. water.

- 241,958.—*System of malting, and apparatus employed therein.* NICHOLAS G. GALLAND.

- 242,015.—*Process of, and apparatus for, distilling turpentine.* JAMES A. MCCREARY.

242,080.—*Dyeing fibrous material.* H. W. VAUGHAN.

“Mechanically incorporating with the fibrous material during the process of manufacture, a mordant, by the aid of infusorial earth or other suitable vehicle for the same, and an oleaginous constituent, and afterward causing the dye-stuff and mordant to chemically combine to form a fast color, by heating or steaming the material.”

*May 31, 1881.*

242,171.—*Process of, and apparatus for, bleaching and purifying liquid bituminous matter.* VICTOR VON OFENHEIM and RUDOLPH VON HAIDINGER.

Filtration through earthy substances.

242,216.—*Process of evaporating, crystallizing and refining saccharine solutions and producing dry refined sugar.* JAMES A. MORRELL.

Cane-juice is subjected to a series of operations in which “deoxidized and electrized” air plays an important part.

242,263.—*Nickel-plating fluid.* GEORGE BRUCKER.

Nitrate of nickel, hyposulphide of soda, and cream of tartar.

242,272.—*Treatment of the saline liquors resulting from soap-manufacture, in order to extract the matters contained therein.* CONSTANT V. CLOUS.

242,337.—*Process of silvering glass.* CONSTANT LAVAL.

Two solutions of nitrate of silver, water, and aqua-ammonia, and of nitrate of silver, water, potassa, and pyroxyline, are mixed and immediately poured upon the glass surface to be silvered.

242,368.—*Process of, and apparatus for, drying air.* GEORGE H. REYNOLDS.

Air is forced, under strong pressure, through water. The latter cools it and takes up the excess of moisture from the air.

*June 7, 1881.*

242,439.—*Saccharification of amylaceous matters by malt.* AUGUSTE P. DUBRUNFAUT.

A process for the production of crystallized maltose from starch with malt, the activity of the latter of which has been increased by “oxidizing its infusion.”

242,546.—*Process of manufacturing and purifying gas for preserving purposes.* CHARLES F., ARTHUR W., and ALBERT L. LAWTON.

Refers to No. 241,677.

242,589.—*Method of treating hominy for preservation.* WILLARD S. BOON.

Hulled corn is wetted with a weak alkaline solution and then dried.

242,624.—*Process of coating metals with lead, zinc, or tin.* WM. FRISHMUTH.

Iron is first nickel-plated, and then immersed into the molten metal.

242,702.—*Apparatus for separating and recovering the fatty matters from bones.* FRIEDRICH SELTSAM.

242,707.—*Azo-color.* JAMES H. STEBBINS, JR.

The product of the reaction of mono-sulpho acid of betanaphthol on a mixture of diazobenzole and diazonaphthalene.

June 14, 1881.

242,783.—*Explosive compound.* JOHN M. LEWIN.

Nitro-glycerine, cellulose, and nitre.

242,855.—*Dyeing fabrics with aniline colors.* NICHOLAS C. ARMAND and JACQUES E. BERTON.

Aniline colors are made soluble in benzine by the admixture of oil.

243,000.—*Method of dyeing leather black.* NIELS G. SOERENSEN.

Tanned leather is treated with a vanadium salt.

June 21, 1881.

243,062.—*Preserved wood.* JOS. H. CONNELLY.

Wood saturated with a solution of sulphate of ammonia.

243,141.—*Dyeing mixed fabrics.* JULES J. LELOIR.

A mordant of a complex composition.

243,178.—*Process of manufacturing an inspissating and sizing paste from animal protéine.* EMIL VON PORTHEIM.

Insoluble albuminoid substances treated with caustic alkali.

243,233.—*Filtering liquids and apparatus therefor.* JOHN F. C. FARQUHAR and WALTER OLDHAM.

243,265.—*Filter.* JOHN W. HYATT.

243,310.—*Process of separating gypsum from the solutions of starch sugar produced by treating the latter with sulphuric acid.* CARL SCHEIBLER.

If a grape-sugar solution, containing sulphate of lime, is boiled with oxalate of barium, two insoluble compounds, oxalate of lime and sulphate of barium, are formed.

June 28, 1881.

243,377.—*Process of manufacturing fatty acids for making candles.* ARMAND MUELLER JACOBS.

243,378.—*Turkey-red mordant.* ARMAND MUELLER JACOBS.

243,428.—*Galvanic battery.* JOSEPH H. CHEEVER.

243,432.—*Explosive compound.* SILAS R. DEVINE.

Chlorate of potash and nitro-benzole.

243,510.—*Process of obtaining perfume of natural flowers and apparatus therefor.* ROBT. A. CHEESEBROUGH.

The absorbing material is placed on trays in a closed compartment which also contains the flowers.

242,635.—*Manufacture of aluminous cake.* CONRAD SEMPER.

Aluminous sulphate, in a semi-fused condition, is treated with zinc-dust.

243,669.—*Method or process of manufacturing glucose from grain.* HORACE WILLIAMS and JOHN L. ALBERGER.

The inventors claim "the process of washing off and eliminating the impurities of crystallized grape-sugar, by pressure or centrifugal force."

243,673.—*Method of treating copper ores containing precious metals.* PAUL G. L. G. DESIGNOLLE.

*July 5, 1881.*

243,685.—*Gelatine prepared in the form of dried foam.* REUBEN BROOKS.

A foam produced from the solution by blowing air into it is dried in a current of dry air.

243,757.—*Manufacture of soap.* PAUL CASAMAJOR.

Ordinary soap is melted, and while it is rapidly agitated, spirits of turpentine or heavy oil of petroleum is added, when the whole combines to a uniform mass, which hardens on cooling.

243,789.—*Method of, and composition for, lining plumbago crucibles.* JOHN PEDDER.

The crucibles have a glaze composed of plumbago, silica, salt, and clay-water.

243,827.—*Manufacture of lactates.* CHAS. E. AVERY.

"Manufacturing lactic acid by the fermentation of a sugar of vegetable origin with a lactic ferment in the presence of nitrogenous matters, chiefly of vegetable origin, and of a substance suitable to gradually neutralize the acid as fast as formed."

243,850.—*Furnace for the reduction of zinc, cinnabar, and other ores.* SAMUEL G. CLARK.

243,910.—*Process of manufacturing distilled spirits from grain.* THOMAS A. and WILLIAM T. JEBB.

Corn is steeped, ground, and beaten. It is then sifted with water and separated into a finely-disintegrated portion which passes through the sieve and into another portion which is retained on the sieve, and which the inventors call "bran and gluten." Only the former is to be used in the manufacture of alcohol.

243,923.—*Tanning process.* ROBERT KOENITZER.

243,986.—*Soap*. ROBERT A. McCULLOUGH.

243,939.—*Grinding and sieving caustic alkali*. WILLIAM G. MENZIES.

It is done while the substance is still hot.

243,949.—*Manufacture of sulphate of alumina*. BENJAMIN E. R. NEWLANDS.

The sulphate of alumina is allowed to crystallize, and the mother-liquor containing the impurities is removed by pressure or centrifugal force.

243,963.—*Treatment of horn shavings*. JULIUS PATHE.

They are treated with boracic and arsenious acids, and then heated to 120° C.

243,991.—*Manufacture of soda*. ERNEST SOLVAY.

“Mixing soda with the bicarbonate of soda previous to the introduction of the bicarbonate into the decomposing apparatus.”

*July 12, 1881.*

244,080.—*Composition for dissolving the coating of gold in ore*. JOHN F. SANDERS.

Cyanide of potassium and phosphoric acid.

244,234.—*Electro-deposition of metal*. PAGET HIGGS.

Refers to the production of aluminium from its borate by galvanic deposition.

*July 19, 1881.*

244,421.—*Means for extinguishing fire in oil-tanks*. CHAS. BARNES.

244,445.—*Process of producing alloys for anodes*. WILLIAM FRISHMUTH.

Refers to the production of an alloy of nickel and aluminium.

244,674.—*Preparing and bleaching jute*. JOSEPH J. SACHS.

*July 26, 1881.*

244,757.—*Coloring matter from tetranitro-naphthol*. EMIL LABHARDT.

By the energetic action of nitric acid on monobromnaphthalene tetranitro-naphthol is produced.

244,788.—*Treating caoutchouc with hydrocarbon oils*. LUDWIG BECKERS.

A solution of caoutchouc in a “hydrocarbon oil” of 250–300° C. boiling point is used for waterproofing.

244,872.—*Method of, and apparatus for, manufacturing artificial leather*. EWALD FISCHER.

244,875.—*Galvanic battery*. JOHN H. A. and ANTON H. B. FOLKERS.

244,902.—*Production of saccharine substances*. S. H. JOHNSON.

The inventor's process of treating grain which has been impregnated with dilute sulphuric acid in a peculiarly constructed converter,



with steam of high pressure, has been found to be also applicable to brewers' or distillers' grains for the production of saccharine solutions therefrom.

*August 2, 1881.*

245,226.—*Distilling, condensing, and refrigerating apparatus.* DÉSIÉ F. SAVALLÉ.

*August 9, 1881.*

245,274.—*Mechanism for, and process of, extracting oil from oleaginous materials.* FRANCIS H. BYERLEY.

245,411.—*Phosphorescent composition.* MERRILL B. SHERWOOD.

Sulphides of calcium combined with carbonate of lime, magnesia, and silic.

245,568.—*Apparatus for testing hydrocarbon fluids.* GEORGE M. SAYBOLT.

*August 16, 1881.*

245,633.—*Process of manufacturing oleaginous mordants.* ARMAND MUELLER JACOBS.

245,661.—*Manufacture of potassium ferrocyanide.* THEODOR RICHTERS.

Nitrogenous substances are impregnated with a solution of carbonate of potassium dried in an atmosphere of carbonic acid and then calcined in retorts. The mass is afterwards boiled in contact with iron.

245,701.—*Thickener for mordants and colors.* JOHN BURTON.

The substance recommended is glucose.

245,750.—*Manufacture of sulphate of alumina.* CONRAD SEMPER.

The impure solution is in the form of a spray treated with sulphurous acid, or sulphuretted hydrogen, in order to reduce ferric sulphate.

245,809.—*Manufacture of sugar.* EMIL FLEISCHER.

Refers to the recovery of sugar from beet-root molasses by means of a bibasic saccharate of strontia.

245,853.—*Ingredient scale.* WILLIAM J. MILLER.

*August 23, 1881.*

245,952.—*Manufacture of plastic compounds from pyroxylyene.* SAMUEL J. HOGGSON and GEORGE C. PETTIS.

The pyroxylyene to be used is treated with sal-ammonia in order to render it less explosive.

245,955.—*Manufacture of soap.* BENNO JAFFÉ and LUDWIG DARMSTAEDTER.

The process of salting soaps with sulphate of soda for the purpose of obtaining a waste lye from which glycerine is produced.

246,073.—*Purifying vegetable oils, and separating them from solvents.* FRANCIS H. BYERLEY.

First steam and then air is blown through the heated oil.

246,096.—*Process of solidifying crude and refined petroleum.* PETER DITMAR.

Two to three per cent. of tallow-soap are to be dissolved in the oil.

246,201.—*Electro-chemical treatment of ores.* EMILE REYNIER.

The object is to recover certain metals from their ores, and at the same time to generate electricity for general application.

246,221.—*Azo-color.* JULIUS SCHUNCKE.

The product of the reaction of diazo-azoxylol upon betanaphthoedisulpho acid, called archil-red.

246,242.—*Treatment of tank waters of slaughterhouses, etc.* BRUNO TERNE.

These waters are to be concentrated, mixed with sulphuric acid, and this mixture used for the treatment of phosphatic substances for the production of a fertilizer.

246,262.—*Method of. and process for, manufacturing glucose from grain.* HORACE WILLIAMS and JOHN L. ALBERGER.

Grain is boiled with very weak acid just long enough to render all the starch soluble. The liquid is then separated by filtration, and by adding more acid and boiling the conversion is completed.

246,272.—*Azo coloring-matter.* HEINRICH BAUM.

The product of the reaction of diazobenzoiesulpho acid upon betanaphtholemonosulpho acid.

*August 30, 1881.*

246,327.—*Methyl-blue color.* CARL A. MARTINS.

The dye-stuff is produced by reducing "methyl-orange No. 3" with sulphohydrate of ammonia, and oxidizing it afterward with perchloride of iron.

246,396.—*Apparatus for concentrating sulphuric acid.* CÆSAR KOLBE and THEODOR LINDFORS.

246,398.—*Method of dephosphorizing crude cast-iron.* LOUIS G. LAUREAU.

246,532.—*Lubricating compounds for car-axles, etc.* GEORGE H. MERRILL.

Tallow, rosin, petroleum-tar, milk of lime, and graphite.

*September 6, 1881.*

246,614.—*Compound hydrocarbon fuel.* JOHN P. JONES.

Infusorial earth impregnated with petroleum.

246,616.—*Desiccating machine.* CORNELIUS KIMPLEN.

246,762.—*Preserving wood.* ERWIN HAGEN.

Uses a solution of chloride of zinc and sulphate of lime in water.

246,793.—*Process of distilling alcohol.* CHARLES W. LAWRENCE.

Adds powdered charcoal to the fermented mash before distilling.

246,816.—*Method of utilizing the residue of the acid treatment of grain and other materials for the manufacture of alcohol.* EUGENE PORION and LOUIS MEHAY.

"Subjecting said residue to pressure in filter-presses and obtaining therefrom both oil and oil cake."

*September 13, 1881.*

246,891.—*Treatment of pyroxylene.* CHARLES S. LOCKWOOD.

It is mixed with a solution of camphor in chloral.

246,900.—*Process of obtaining pure juice from sugar-cane.* WILLIAM A. MARTIN.

The cane properly sliced is treated with steam in a closed vessel, and the remaining juice washed out with water.

246,908.—*Process of producing a metal high in phosphorus and carbon and low in silicon.* JACOB REESE.

246,949.—*Apparatus for the continuous distillation of ammonia.* HERMANN GRUENEBERG and JACOB GAREIS.

246,958.—*Polishing composition.* ALEXANDER LEVETT.

Uses glucose as a base for holding the polishing materials.

247,046.—*Process of, and apparatus for, making ammonium sulphate.* HUBERT GROUVEN.

247,083.—*Process of manufacturing carbons.* HIRAM S. MAXIM.

Heats carbonized strips to be used in electric lamps by an electric current in an atmosphere of naphtha vapor in order to stiffen and strengthen them.

*September 20, 1881.*

247,152.—*Process of treating corn in the manufacture of starch, glucose, and other products therefrom.* ARNO BEHR.

This process is based on the fact that, by a proper treatment of steeping, crushing, and disintegrating, the germs of corn are obtained almost unbroken and free from the mealy parts and the hulls. If then sufficient water is added to bring the mixture of meal and water to about 10° Bé density, the germs rise to the top while the hulls sink. The process is conducted in a special apparatus which effects a continuous separation of the substance of the corn into germs, hulls, and mealy parts.

247,271.—*Method of making lime brick.* JACOB REESE.

Uniting the particles of indurated lime by the use of a fluid silicate of lime.

247,288.—*Process of manufacturing sugar.* JOHN L. ALBERGER.

Corn is treated with water and steam under pressure until all the starch is liquified. The solution is separated from the insoluble residue by filtration, and then converted in the usual way.

247,388.—*Method of filtration.* GEORGE F. MEYER.

Filtration of saccharine solutions through granular mineral substances instead of through charcoal.

247,455.—*Method of treating grape-sugar or glucose for the purpose of refining the same.* HORACE WILLIAMS and JOHN L. ALBERGER.

The inventors propose to eliminate the bitter and other impurities of raw grape-sugar solutions by fermentation. The specification omits to state how this fermentation is to be conducted.

*September 24, 1881.*

247,494.—*Hardening copper.* ANNIE GETCHELL.

Addition of a compound of potash, alum, bone-dust, and zinc.

247,804.—*Centrifugal creamer.* GUSTAF DE LAVAL.

247,834.—*Manufacture of hard rubber.* THOMAS J. MAYALL.

Mixing pure rubber with sulphide of antimony, and curing the compound by heat.

247,957.—*Refining starch sugar.* FRANZ SOXHLET.

The process of refining consists in treating impure grape-sugar with, or recrystallizing it from, methyl or ethyl alcohol, and removing the alcoholic solution containing the impurities, by pressure or centrifugal force. If a concentrated solution of the purified grape-sugar is allowed to solidify at a temperature above 30° C., hydrate of grape-sugar of a distinct crystalline structure is obtained.

247,958.—*Refining and crystallizing starch sugar.* FRANZ SOXHLET.

Crystallized anhydrous grape-sugar is produced from a solution of grape-sugar in methylic alcohol.

*October 11, 1881.*

248,041.—*Yeast.* WILLIAM H. HIGGINS.

Adds glycerine to the mixture of yeast and starch.

248,153.—*Process of preparing leuco-base of aniline-blue.* OTTO FISCHER.

The process consists in making para-amidobenzaldehyde react upon a mixture of diphenylamine and methyldiphenylamine in the presence of chloride of zinc.

248,154.—*Process of preparing coloring matter.* OTTO FISCHER.

If para-amidobenzaldehyde is made to react upon a mixture of aniline and toluidine the leuco-base of rosaniline is produced.

248,183.—*Apparatus for meteorology.* WILHELM KLINKERFUES.

248,246.—*Coloring matter.* OTTO N. WITT.

A violet dye formed by the action of nitrosodimethylaniline on metaphenylenediamine.

*October 18, 1881.*

248,313.—*Glucose.* FRANK HIGEL.

The root of the plant manihot is used for the manufacture of glucose.

248,314.—*Manufacture of soap.* CHARLES S. HIGGINS.

248,343.—*Process of, and apparatus for, cleansing meal from which oil has been extracted.* RAYMOND S. PERRIN.

248,413.—*Compound of celluloid and luminous material.* ISAAC W. DRUMMOND.

Celluloid and sulphide of calcium.

248,425.—*Apparatus for producing high vacuums.* THOMAS A. EDISON.

248,431.—*Preserving fruit.* THOMAS A. EDISON.

The fruit is placed in a glass vessel which is sealed by fusion after a high vacuum has been produced.

*October 25, 1881.*

248,606.—*Apparatus for collecting waste hydrogen in the manufacture of iron wire.* RICHARD N. R. PHELPS.

248,632.—*Process of purifying gas.* FRANCIS J. BOLTON and JAMES A. WANKLYN.

Dry superphosphate is used in which the free acid has been neutralized with ammonia-water.

248,654.—*Secondary electric battery.* PAUL JABLOCHKOFF.

248,674.—*Manufacture of annealed, hardened, and toughened glass articles.* FREDERICK SIEMENS.

248,768.—*Filter.* JUAN F. N. MACAY.

248,769.—*Electrophore, or secondary battery.* JAMES A. MALLONEY.

248,797.—*Method of preserving milk.* GUSTAV ROTHE.

The caseine is coagulated by addition of lactic acid, the clear liquid evaporated to dryness and mixed with the dried caseine.

248,800.—*Apparatus for treating mineral and other substances with acids.* AMÉDÉE M. G. SÉBILLOT.

248,833.—*Compound for the desulphuration of liquids and gases.* FRIEDRICH LUX.

“Artificial peroxide of iron combined with alkalies.”

*November 1, 1881.*

248,904.—*Gum substitute.* GEORGE F. WILSON and PHILIP O'REILLY.

In the manufacture of dextrine from starch, hydrochloric acid is used instead of nitric acid, and the material kept flowing in a continuous stream and agitated while being roasted.

248,972.—*Manufacturing glucose or grape-sugar.* HORACE WILLIAMS and JOHN L. ALBERGER.

"Grinding the corn wet, then washing and separating the starch from the husk, then taking it directly from the separator to the cooker, converting it into dextrine and filtering it before saccharification."

249,136.—*Production of coloring matter.* OTTO N. WITT.

A dark-red dye formed by the action of nitrosodimethyl aniline on metatoluylenediamine, belonging to a class of coloring matters called indophenols by the inventor.

*November 8, 1881.*

249,161.—*Apparatus for making vinegar.* JAS. CUSHING.

249,420.—*Process for manufacturing phosphorescent substances.* ORLANDO THOWLESS.

*November 15, 1881.*

249,490.—*Explosive compound.* CARL W. VOLNEY.

A mixture of monochlordinitrine and nitrate of potassium.

249,506.—*Manufacture of soap.* MIGUEL DE LA VEGA and LUIZ D'OLIVEIRA.

Castor-oil soap.

249,548.—*Preparing, indurating, and solidifying calcareous lining materials for furnaces.* JACOB REESE.

Melts the carbonates of lime or magnesia under sufficient pressure to prevent the escape of the carbonic acid.

249,701.—*Explosive compound.* THOMAS VARNEY.

249,731.—*Apparatus for separating fluids.* GUSTAF DE LAVAL.

*November 22, 1881.*

249,856.—*Preserving wood.* SAMUEL R. PERCY.

A solution containing a resinous gum and picric acid is used.

249,936.—*Coloring matter.* JOHANNES H. H. O. GUERKE.

"Produced by the action of the disulphobetanaphtholic sodium salt, insoluble in alcohol, on the diazoic derivative of para-amidocinnamic acid methylic ether."

250,009.—*Lubricating compound.* EDMUND SMALLEY.

Tallow, pine-tar, and sulphur.

250,035 and 250,036.—*Manufacture of artificial indigo.* ADOLPH BAEYER.

Orthonitrophenylpropionic ether, on reduction with sulphhydrate of ammonium, gives indogenic ether, from which indogenic acid can be obtained by treatment with caustic alkali. Indogenic acid, under the influence of heat, is transformed into indogen. Both these substances can be used for the production of indigo-blue.

250,038.—*Manufacture of crimson coloring-matter.* HEINRICH BAUM.

It is produced "by the action of the disulpho betanaphtholic sodium salt, insoluble in alcohol, on the diazoic derivative of amido-para-creosote-methylic ether."

*November 29, 1881.*

250,071.—*Process of manufacturing albumen.* UNO H. HILLMANN.

Crushes fish-spawn with water and evaporates the solution under a vacuum at a temperature low enough to prevent coagulation.

250,117.—*Manufacture of glucose.* HENRY A. WEBER and MELLVILL A. SCOVELL.

The application of the seeds of the sorghum plant to the manufacture of starch and sugar.

250,118.—*Process of manufacturing sugar and syrup.* HENRY A. WEBER and MELVILL A. SCOVELL.

The process refers to the treatment of sorghum-juice, which consists in exactly neutralizing the juice with lime and, after boiling and defecating, filtering it through bone-black.

250,201.—*Sulphonated compound of rosaniline.* HEINRICH CARO.

Trisulpho rosaniline produced by the action of crystallizable sulphuric acid on rosaniline.

250,247.—*Manufacture of rosaniline colors.* JOHN HOLLIDAY.

Another sulpho-conjugated compound of rosaniline.

250,326.—*Inoxidizable alloy.* PETER DE VILLIERS.

An alloy of tin, lead, and silver.

*December 6, 1881.*

250,333.—*Process of manufacturing crystallized anhydrate of grape-sugar from a watery solution of grape-sugar.* ARNO BEHR.

From concentrated solutions of high purity grape-sugar crystallizes in the anhydrous form, and not, as was generally believed, as a hydrate. If this crystallization is properly conducted and the liquid part removed by means of a centrifugal machine, crystallized anhydrous grape-sugar in the form of blocks resembling hard cane-sugar in appearance can be produced.

250,334.—*Method of refining grape-sugar.* ARNO BEHR.

Even in a concentrated solution of ordinary grape-sugar of moderate purity a crystallization of anhydrous grape-sugar can be induced if a very small quantity of crystallized anhydrous grape-sugar is stirred into the solution. On this observation is based a process for making from the product of the conversion of starch a grape-sugar of great purity, by crystallizing it and removing the impure liquid by means of a centrifugal machine.

250,416.—*Manufacture of sulphuric acid.* FREDERICK BENKER and HENRI LASNE.

“Mixing sulphurous acid gas with the gases which enter the Gay-Lussac tower.”

250,418.—*Apparatus for filtering liquids.* FRANÇOIS A. BONNEFIN.

250,524.—*Process of, and machinery for, refining paraffine and other waxy matter.* DANIEL T. GRAY.

250,605.—*Manufacture of white lead.* WILLIAM THOMPSON.

Acetic acid vapor and air, and afterwards carbonic acid and air, are blown over sheet lead contained in a closed chamber.

250,824.—*Mode of making sugar.* WILLIAM LITTLEJOHN.

The defecation of the juice with lime and the bleaching with bisulphite of lime, shall be conducted in the following new and peculiar manner, viz. : Successive additions of these chemicals shall be made, and after each addition it shall be ascertained with a small sample, “by ocular test,” if a further addition produces an improvement.

*December 20, 1881.*

251,145.—*Explosive compound.* GUSTAV VON PLANITZ.

The product of a treatment of rosin with nitric acid.

251,162—251,164.—*Dye-stuff or coloring matter.* HEINRICH BAUM.

The first is the product of the reaction of the disulpho betanaphtholic sodium salt, soluble in alcohol, on diazobenzol; the last, of a mixture of two disulpho-betanaphtholic acids upon diazonaphthalene; the middle number only mentions a deep-red scarlet dye-stuff.

251,187.—*Zinc pigment, and process of making the same.* JOHN CAWLEY.

Precipitated sulphide of zinc and sulphate of barium are intimately mixed with caustic magnesia, calcined and ground.

251,279.—*Manufacture of paint for ships' bottoms, etc.* SAMUEL R. PERCY and HENRY A. MOTT, JR.

A paint containing picric acid or its salts.

*December 27, 1881.*

251,391.—*Manufacture of friction matches.* LOUIS WAGNER.

Gives the following proportions of a composition for topping matches : Hyposulphite of lead 34, peroxide of lead 5, chlorate of pot.



ash 16, gray sulphuret of antimony 6, pulverized charcoal 5, pulverized glass 10, saltpetre 4, sulphur 1, dextrine  $1\frac{1}{4}$ , Cologne glue  $1\frac{1}{4}$ , water 30 parts

251,473 and 251,474.—*Insulating composition or compound for coating electric or other wires and conductors.* FREDERICK W. SCHROEDER.

Glue, mastic, dextrine, chrome-alum, chloride of iron, and glycerine, with or without asbestos and albumen.

251,499.—*Manufacture of artificial indigo.* ADOLPH BAEYER.

The claim is for a new derivative of orthonitrophenylpropionic acid called orthonitro-acetenylbenzene, from which indigo can be obtained.

251,500.—*Manufacture of artificial indigo.* ADOLPH BAEYER.

Another substance from which indigo can be obtained is di-isatogen, made from orthodinitro-acetenylphenyl with fuming sulphuric acid.

251,501.—*Manufacture of soluble derivatives of indigo-blue.* ADOLPH BAEYER.

Indigo-white sulphuric acid is produced by heating a mixture of indigo, ferrous sulphate, caustic potash, and an alkaline pyrosulphate.

251,671.—*Preparation of new material for the manufacture of artificial indigo.* ADOLPH BAEYER.

This material is orthodinitrodiacetenylphenyl, prepared from orthonitro-acetenylbenzene by a process which cannot be here described.

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## Foreign Patents

Condensed from R. BIEDERMANN'S Report to the German Chemical Society,

by OTTO H. KRAUSE.

J. W. KLINGHAMMER, Braunschweig : *Preparation of saccharate of lime from "Melassekalk" \* by means of paraffine oil and other unsaponifiable oils of high boiling points.* (Germ. P., No. 11365, February 28, 1880.)—The syrup lime is treated successively with water of 80°–90° C. and paraffine oil of 100°–120°; the latter displaces the lye formed by the water in from 2 to 3 hours. The oil dissolves organic bases contained in the syrup lime. Then water of 80°–90° C. in its turn displaces the oil. Finally, the oily syrup lime is mixed with one-sixth of its bulk of water and heated by steam, whereby it melts, the oil forming a layer on the surface of the liquid. After syphoning it off and treating with acid, to obtain the organic bases, it can be used over again. Instead of the water, very dilute alcohol may be employed.

O. H. KRAUSE, Jersey City : *Improvements in apparatus for separating liquids from solid matters by means of centrifugal force.* (Germ. P., No. 11778, January 24, 1880.)—Consists of suitably shaped vessels of metal, glass, porcelain, etc., which

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\* *Melassekalk*.—Syrup lime is an impure saccharate of lime and is made by mixing the low grade syrups of beet-sugar factories and refineries with sufficient powder of slaked or unslaked lime to form a dry, porous mass.—O. H. K.

can be tightly closed and placed into the revolving basket of a centrifugal machine. Each vessel is divided into two compartments by means of a perforated vertical diaphragm and an inclined wall; the latter does not quite extend to the top. The chamber next to the center of the basket is filled with the mixture to be operated upon. The outer one receives and retains the liquid which is driven into it by centrifugal action, the inclined wall preventing its return to the dried substance. See also this JOURNAL, 1, 506.

I. BÖNNY, Stulberg : *Gas-meter for chemical analyses*. (Germ. P., No. 12360, June 1, 1880.)—This apparatus, which is intended for aspirating and measuring large quantities of gas, consists of a measuring vessel containing a syphon, and is suspended by means of a spiral spring over a vessel of water having a constant level, under the surface of which the longer leg of the syphon passes. The measuring vessel is so connected with the service pipes as to carry on its functions automatically.

G. BORSCHÉ and I. BRÜENJES, Leopoldshall : *Improvements in the method of working kainite*. (Germ. P., No. 12875, April 17, 1880.)—Relates to improvements upon Germ. P., No. 10701. See this JOURNAL, 2, 406.

HUGO MÜLLER, Kohlscheid : *Manufacture of aluminic hydrate and alkalies from native aluminic silicates*. (Germ. P., No. 12947, April 13, 1880.)—Aluminic silicates are intimately mixed with limestone, magnesite, or dolomite, and then with alkaline hydrates, carbonates, etc., and heated to bright redness. From the alkaline aluminate extracted from the mass, aluminic hydrate and alkaline carbonates are obtained in known manner.

BADISCHE ANILIN UND SODAFABRIK, Ludwigshaven : *Production of coloring matters by the action of diazo-anisols upon naphthols and naphtholsulphonic acids*. (Germ. P., No. 12451, Jan. 3, 1879.)—The amido derivatives of phenolic ethers are converted into diazo compounds, and then combined as such, or in the form of sulphonic acids, with amines or phenols to produce azo-dyes.

K. OEHLER, Offenbach : *Method of obtaining coloring matters by means of Lauth's reaction*. (Germ. P., No. 12932, July 14, 1880.)—Two reactions for obtaining amido-ethylaniline are described. The latter compound is treated, after the method given by Lauth, with hydrogen sulphide and oxidizing agents (ferric chloride), when it behaves like phenylenediamine, and yields a blue coloring matter.

C. M. WARREN, Paris : *Method of obtaining anthracene from coal-tar*. (Germ. P., No. 12933, July 16, 1880.)—Residues from the distillation of coal-tar are mixed with petroleum residues and again distilled. The method for separating the anthracene from the paraffines obtained at the same time is described.

A. DOMEIER and C. J. MARZELL, London : *Method of preparing artificial alizarine*. (Germ. P., No. 12938, Aug. 11, 1880.)—Relates to the treatment of the solution of the alizarine-melt in hot water with sulphurous acid, and to its recovery as well as that of the alkalies from the filtrate by means of causticizing with lime. The sulphuric acid required for decomposing the resulting calcium sulphate is obtained as a by-product in the preparation of anthraquinone.

N. G. SØRENSEN, Stockholm: *Black dye for leather*. (Germ. P., No. 13185, Oct. 13, 1880.)—A 1 per cent. solution of ammonium vanadate.

P. RICHTER and W. TRIEBEL, Berlin: *Apparatus for carburetting air*. (Germ. P., No. 12150, June 22, 1880.)—An apparatus for impregnating air with gasolene vapor, combined with an automatic regulator, by means of which a uniform carburation of the air is attained.

F. LITTMANN, Halle: *Ice generator and cooling apparatus*. (Germ. P., No. 11970, Feb. 7, 1880.)—The drum containing the cooling medium revolves upon its axis within the vessel containing the water to be frozen, and touches the surface of the latter. When an ice crust of sufficient thickness has formed, the drum is turned half round, and the ice thawed off by passing a warm current through the drum.

I. TOWNSEND, Stassfurt: *Improvements in the method of preparing chlorine*. (Germ. P., No. 12885, June 22, 1880.)—Heated air is passed through 40°–50° Bè solution of magnesium chloride, to which 10 per cent. of manganic oxide have been added.

ACTIENGESSELLSCHAFT CROIX, Croix: *Improvements in the method of obtaining alkaline carbonates by means of trimethylamine*. (Germ. P., No. 13397, July 10, 1880, 2d addition to Germ. P., No. 5786. See this JOURNAL, 1, 298.)—Sodium sulphate is employed instead of the chloride. Its conversion into bicarbonate results when the apparatus is charged with 1 eq. of sodium sulphate and 1½ to 2 eq. of trimethylamine, and carbonic acid is passed in to saturation. By adding milk of lime to the trimethylamine sulphate, and distilling, the base is recovered.

W. J. WILLIAMS, Chester: *Manufacture of phosphates*. (Eng. P., No. 993, March 8, 1880.)—Native phosphates of iron, alumina, or the alkaline earths, are pulverized, intimately mixed with sodium chloride, and then with finely powdered coke, coal, or other reducing agent. The mixture is heated in a retort, and air and superheated steam passed over the mass which is kept stirred. Hydrochloric acid is evolved and sodium phosphate and aluminate formed.

T. TWYNAM, London: *Manufacture of magnesia*. (Eng. P., No. 1019, March 9, 1880.)—Pulverized dolomite is digested and boiled with solution of calcium chloride. From the filtrate separated from the magnesia calciumoxychloride crystallizes upon cooling. The latter is decomposed by cold water into calcium chloride and calcium hydrate. See Closson's patent, this JOURNAL, 2, 461.

A. CHAPMAN, Liverpool: *Apparatus for recovering soda from waste waters*. (Eng. P., No. 1078, March 12, 1880.)—Intended for recovering the soda from the very dilute solution in which esparto grass is boiled in paper mills. It consists of a steam boiler, into which the water is fed to be concentrated, a battery of vacuum pans, calcining furnace, etc., and an arrangement for utilizing the waste heat from the latter.

RAOUL BRULIÉ and A. LECLERC, Paris: *Obtain ammonia and ammoniacal salts from urine* (Eng. P., No. 1086, March 13, 1880), by adding milk of lime to the liquid and passing it in thin layers over heated surfaces in a distilling apparatus.

H. WIGGIN & Co., Birmingham : *Treatment of nickel and cobalt.* (Eng. P., No. 1058, March 11, 1880.)—From 2 to 5 per cent. of pure manganese or ferromanganese are added to the molten metals to increase their ductility and malleability.

J. E. REYNOLDS, Dublin : *Method of coating metals with lead sulphide or lead luster.* (Eng. P., No. 1112, March 15, 1880.)—The objects to be coated may be of metal, glass, leather, paper, and the like ; they are immersed in an alkaline solution of lead nitrate or tartrate, to which a slowly-decomposing sulphur compound has been added. As such the inventor employs sulphurea. The deposition takes place at 70° C.

W. MAJERT, Elberfeld : *Method of preparing blue coloring matters.* (Germ. P., No. 13281, April 13, 1880.)—A solution of 1 molecule of hydrochloride of nitrosodimethylaniline in 20 times its weight of hydrochloric acid, is mixed with the solution of 1 molecule of sodium thiosulphate dissolved in 10 parts of water. It is slowly heated. Upon cooling, an oxidizing agent is added when the coloring matter forms.

L. BECKERS, Spandau : *Method of dissolving caoutchouc in hydrocarbons of high boiling points.* (Germ. P., No. 13307, June 27, 1880.)

E. MOURLOT, FILS, Paris : *Substitute for gutta-percha.* (Germ. P., No. 13332, August 4, 1880.)—A decoction of birch-bark, evaporated to a thick paste, soon becomes hard in the air and behaves like caoutchouc.

T. H. GRAY, London : *Improvements in the treatment of oils for the manufacture of varnish.* (Germ. P., No. 12825, Jan. 10, 1880.)—Heated air is passed through linseed oil for several hours at a temperature of 120° C., and an equal length of time afterwards at 205° C. When acid vapors cease to be evolved, the oil suddenly becomes thick and forms a light gelatinous mass.

T. L. ALEMAND, Paris : *Substitute for leather.* (Eng. P., No. 1077, March 12, 1880.)

S. CLIFT, Conway, Wales : *Manufacture of coal-tar products.* (Eng. P., No. 967, March 5, 1880.)—Relates to the recovery of the alkali employed in purifying the oils.

A. GAWALOWSKI, Traritzsch, and F. TEICHMANN, Wagstadt : *Method of refining cane-juice, muscovadoes, and syrups.* (Eng. P., No. 861, Feb. 27, 1880.)—The inventors claim that by adding from 0.2 to 0.5 per cent. of aluminium phosphate to the heated liquor, filtration over bone-black is rendered unnecessary. The aluminium phosphate is prepared by dissolving aluminium hydrate in phosphoric acid, or from a mixture of bone-black dust, sulphuric acid, and aluminium sulphate. They also claim the finely divided carbon resulting from the manufacture of potassium ferrocyanide and from the calcination of nitrogenous organic matters.

M. HONIGMANN, Grevenberg : *Apparatus for distillation and absorption.* (Germ. P., No. 12889, July 18, 1880.)—An inclined cylinder, containing a large number of perforated diaphragms through which the gases and vapors pass in an opposite direction to the condensing liquid.

WEGELIN and HUEBNER, Halle : *Vacuum apparatus, with a separate vessel for the evaporating liquid.* (Germ. P., No. 12297, March 12, 1880.)

RHEINISCHE STAHLWERKE, Ruhrort, and HEVERDER BERGWERKS UND HUETTENVEREIN, Hoerde : *Method of dephosphorizing iron in the Bessemer process.* (Germ. P., No. 12700, April 10, 1880.)—Lime or dolomite, or a mixture of 8 parts lime and 1 part ferric oxide, is put into the converter before the metal is run in. The weight of this flux is about equal to double the amount of phosphorus and silicium contained in the charge. The rest of the specification relates to the manner of working the converter.

T. FLEITMANN, Iserlohn : *Method of welding iron, steel, copper and alloys of the latter, with nickel, cobalt and their alloys.* (Germ. P., No. 13304, June 8, 1880.)—Complete exclusion of air from the surfaces to be welded is necessary. To attain this the metals are enclosed in thin sheet iron, which is subsequently removed, or the metals are heated in air-tight vessels, or the air in the furnace is displaced by carburetted hydrogen or carbonic oxide.

I. GARNIER, Paris : *Manufacture of nickel alloys.* (Eng. P., No. 3009, July 21, 1880.)—Pure nickel, after melting, contains occluded oxygen, which renders it brittle. By adding phosphorus or phosphide of nickel to the metal the deleterious action of oxygen is prevented.

P. DE VILLIERS, St. Leonard : *Non-oxidizable alloys.* (Eng. P., No. 8394, Aug. 20, 1880.)—To be used chiefly for coating other metals, and is composed of 80 parts tin, 18 parts lead, and 2 parts silver.

B. BOLLINGER, Milan : *Infusible materials for lining furnaces, converters, etc.* (Eng. P., No. 3393, Aug. 20, 1880.)—Asbestos or serpentine, or mixtures of them, are mixed to a plastic mass with silicate of soda.

K. and T. MOELLER, Kupferhammer, near Brackwede : *Apparatus and methods for purifying feed water for steam boilers.* (Germ. P., No. 12496, Feb. 12, 1880 ; first addition to Germ. P., No. 7343 ; see this JOURNAL, I, 503.)—Reference must be had to the original specification, in which the plant is minutely described and illustrated by drawings.

C. OTTO, Dahlhausen : *Arrangement for heating the gases from coking furnaces after tar and ammonia have been separated from them.* (Germ. P., No. 13156, September 14, 1880.)

A. PONGOWSKY, Sorgues sur l'Ouzève : *Apparatus and method for recovering the light hydrocarbons from petroleum, which are retained by the substances from which oil has been extracted.* (Germ. P., No. 12347, Feb. 29, 1880.)—Warmed air is passed through the substances. The air, charged with hydrocarbons, passing out of the apparatus is led to condensers.

G. W. DAVEY, Barking : *Distillation of coal-tar.* (Eng. P., No. 2666, June 29, 1880.)—Heated air is injected into the still.

I. P. STRENG, Fuerth : *Preparation of odorless brewers' pitch.* (Germ. P., No. 12811, May 30, 1880.)

**E. N. HORSFORD**, Cambridge : *Pulverulent preparations of phosphoric acid for the manufacture of baking powder.* (Eng. P., No. 2875, June 12, 1880.)—A concentrated solution of phosphoric acid is mixed with starch, dried and pulverized. By mixing this with a dry alkaline carbonate a baking powder is formed.

**F. R. MALLET**, Calcutta : *Paper filters.* (Eng. P., No. 2961, July 17, 1880.)—Instead of folding the filters from sheets, they are manufactured into a single conical piece by coating the inner surface of a porous cone with the paper pulp, and applying suction.

**R. S. NEWALL** and **F. S. NEWALL**, Newcastle : *Apparatus for effecting chemical decompositions.* (Eng. P., No. 2417, June 15, 1880.)—This invention relates chiefly to the decomposition of salt in the manufacture of soda. The sulphate pans are of the common form, but made of phosphor-bronze, and are jacketed so that steam or heated air can be applied to them. The mixture of acid and salt is kept in motion by means of a peculiar stirring apparatus. They are also supplied with arrangements for working them continuously.

**I. P. RICKMAN** and **J. B. THOMPSON**, London : *Manufacture of ammonia.* (Eng. P., No. 896, Mar. 1, 1880.)—The ammonia is generated by the slow combustion of coal in air and steam. Pyrites can be mixed with the coal when ammonium sulphate is to be made. The products of combustion are drawn, by means of an exhauster and bent tubes, through the absorbing liquid contained in a series of cells.

**W. C. YOUNG**, Poplar, Middlesex : *Manufacture of ammonium sulphate.* (Eng. P., No. 1310, Mar. 30, 1880.) *Manufacture of ammonium sulphate.* (Eng. P., No. 1310, Mar. 30, 1880.)—The products of the distillation of gas liquor are passed through a furnace together with a current of air, whereby the ammonium sulphide is converted into sulphite, and partly, also, into sulphate. They are then passed into condensers. To convert all the ammonium sulphite contained in the solution into sulphate, heated air is passed through the liquid.

**T. G. YOUNG**, Kelly : *Preparation of ammonia.* (Eng. P., No. 1640, April 26, 1880.)—Electric discharges are passed through a vessel containing 3 volumes of hydrogen and 1 volume of nitrogen.

**W. A. HILLS**, Saltney : *Preparation of dicalcium and tricalcium phosphate.* (Eng. P., No. 1512, April 13, 1880.)—Proposes to decompose sodium phosphate by means of calcium thiosulphite obtained from alkali waste.

**C. C. WALKER**, Lilleshall, and **W. T. WALKER**, Highgate : *Method and apparatus for purifying illuminating gas.* (Germ. P., No. 2059, April 2, 1880.)—A cylinder, containing a number of diaphragms of gradually diminishing size of mesh, is placed between the condenser and scrubber for the purpose of retaining particles of tar.

**PAUL AUBE**, Paris : *Apparatus for the simultaneous production of steel and illuminating gas.* (Germ. P., No. 12837, April 15, 1880.)—A retort is charged with alternate layers of charcoal or coke and iron. A current of steam superheated in the same furnace is passed through this mass. At the same time, fatty matters are made to flow along the bottom of the retort which is heated to redness.

CHEMICAL WORKS "EISENBUETTTEL," Braunschweig: *Preserving salt*. (Germ. P., No. 13545, May 28, 1880.)—Nitric acid and common salt are added to a fused mixture of 4 equivalents of boracic acid and 1 equivalent of sodium phosphate.

CH. LOWE and JOHN GILL, Manchester: *Coal tar manufactures*. (Eng. P., No. 1456, April 9, 1880.)—Phenols are obtained from the tar oils by decomposing the alkaline lyes with which they have been treated by means of sulphurous acid.

R. PUNSHON, Brighton: *Explosive compound*. (Eng. P., No. 2242, June 1, 1880.)—Nitric acid is mixed with pulverized asbestos and picric acid. The doughy mass is put into paper capsules and coated with a cement of powdered glass and silicate of soda.

C. A. BURGHARDT, Manchester: *Recovery of valuable products from fatty matters*. (Eng. P., No. 3030, July 23, 1880.)—Relates to the treatment of fatty matters containing alizarine, from dye works. The mass is distilled at 220° C. upon a sand or glycerine bath, and the fats or stearine passing over collected. The residue is extracted with methylated spirits.

S. SPENCE, Manchester: *Purification of waste waters*. (Eng. P., No. 2227, June 1, 1880.)—After the sewerage has been partially clarified it is mixed with a solution of aluminic or ferro-aluminic chloride or sulphate. As soon as the flocculent precipitate has settled the clear and odorless water can be drawn off. The alumina and ferric-oxide may be recovered by treating the precipitate with sulphuric acid in a separate vessel.

P. GONDOLO, Paris: *Extraction of tannin*. (Germ. P., No. 12513 and No. 12876, of July 2, and August 23, 1880, respectively. Additions to Germ. P., No. 7864; see this JOURNAL, 2, 103.)—By treating the substances containing tannin with solutions of alkaline sulphites or bisulphites the deleterious lime salts are removed. The extract which contains sodium tannate is mixed with acid, clarified and evaporated. In Germ. P., No. 12513, the apparatus employed by the inventor is described. It is arranged and manipulated like a diffusion battery.

I. B. MACKAY, London: *Manufacture of a soluble cerium compound for medicinal purposes*. (Eng. P., No. 1275, March 25, 1880.)—A feebly alkaline solution of ammonium—cerium nitrate.

E. SOLVAY, Brussels: *Decomposition of calcium chloride for the preparation of hydrochloric acid and chlorine*. (Eng. P., No. 838, Feb. 25, 1880.)—A mixture of calcium chloride and clay is heated in a kind of cupola furnace and decomposed by means of steam or air, as the case may be. The calcium silico-aluminate resulting from this process yields a good cement when mixed with a small amount of lime, and calcined. For this purpose it is necessary that the decomposition of the calcium chloride be effected at as low a temperature as possible. (Eng. P., No. 840, Feb. 25, 1880.)

E. SOLVAY, Brussels: *Manufacture of chloride of lime*. (Eng. P., No. 837, Feb. 25, 1880.)—Slaked lime in the form of small pellets is placed in a cylinder into which chlorine enters from below. The latter is completely absorbed, and the chloride of lime obtained in the form of pieces or balls.



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CHEMICAL WORKS "EISENBUETEL," Braunschweig: *Preserving salt*. (Germ. P., No. 13545, May 28, 1880.)—Nitric acid and common salt are added to a fused mixture of 4 equivalents of boracic acid and 1 equivalent of sodium phosphate.

CH. LOWE and JOHN GILL, Manchester: *Coal tar manufactures*. (Eng. P., No. 1456, April 9, 1880.)—Phenols are obtained from the tar oils by decomposing the alkaline lyes with which they have been treated by means of sulphurous acid.

R. PUNSHON, Brighton: *Explosive compound*. (Eng. P., No. 2242, June 1, 1880.)—Nitric acid is mixed with pulverized asbestos and picric acid. The doughy mass is put into paper capsules and coated with a cement of powdered glass and silicate of soda.

C. A. BURGHARDT, Manchester: *Recovery of valuable products from fatty matters*. (Eng. P., No. 3030, July 23, 1880.)—Relates to the treatment of fatty matters containing alizarine, from dye works. The mass is distilled at 220° C. upon a sand or glycerine bath, and the fats or stearine passing over collected. The residue is extracted with methylated spirits.

S. SPENCE, Manchester: *Purification of waste waters*. (Eng. P., No. 2227, June 1, 1880.)—After the sewerage has been partially clarified it is mixed with a solution of aluminic or ferro-aluminic chloride or sulphate. As soon as the flocculent precipitate has settled the clear and odorless water can be drawn off. The alumina and ferric-oxide may be recovered by treating the precipitate with sulphuric acid in a separate vessel.

P. GONDOLO, Paris: *Extraction of tannin*. (Germ. P., No. 12513 and No. 12876, of July 2, and August 23, 1880, respectively. Additions to Germ. P., No. 7864; see this JOURNAL, 2, 103.)—By treating the substances containing tannin with solutions of alkaline sulphites or bisulphites the deleterious lime salts are removed. The extract which contains sodium tannate is mixed with acid, clarified and evaporated. In Germ. P., No. 12513, the apparatus employed by the inventor is described. It is arranged and manipulated like a diffusion battery.

I. B. MACKAY, London: *Manufacture of a soluble cerium compound for medicinal purposes*. (Eng. P., No. 1275, March 25, 1880.)—A feebly alkaline solution of ammonium—cerium nitrate.

E. SOLVAY, Brussels: *Decomposition of calcium chloride for the preparation of hydrochloric acid and chlorine*. (Eng. P., No. 838, Feb. 25, 1880.)—A mixture of calcium chloride and clay is heated in a kind of cupola furnace and decomposed by means of steam or air, as the case may be. The calcium silico-aluminate resulting from this process yields a good cement when mixed with a small amount of lime, and calcined. For this purpose it is necessary that the decomposition of the calcium chloride be effected at as low a temperature as possible. (Eng. P., No. 840, Feb. 25, 1880.)

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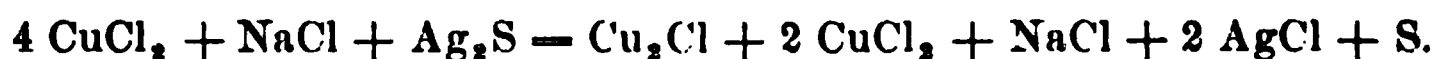
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1. Silver sulphide, if present, is thereby converted into chloride.



After filtration the residue is washed with hot acidified water to remove copper salts. It is then treated with sodium thiosulphate to dissolve chloride of silver. The residue is now sulphur and gangue.

The solution of AgCl in sodium thiosulphate is boiled with sulphur to precipitate AgS and recover the thiosulphate. The AgS is boiled with CuCl<sub>2</sub> and

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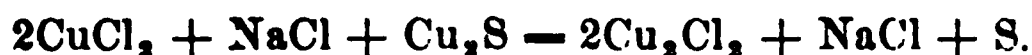
The AgCl in the NaCl solution is precipitated in the metallic state by means of iron. The ferric chloride is worked up into colcothar. (See Pat., No. 9565; this JOURNAL, 2, 192.)

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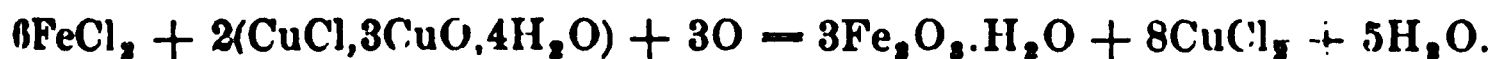
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4. When galena is to be treated, the before mentioned process being employed, we get



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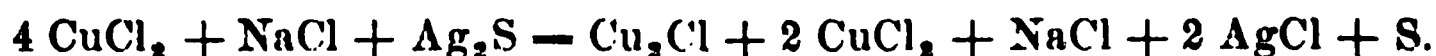
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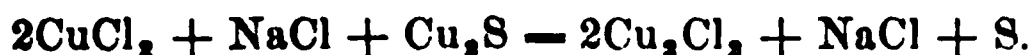
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S. J. COX, Yatton, Somerset: *Tanning hides*. (Eng. P., No. 2839, July 9, 1880.)—The prepared hides are first washed with dilute tanning liquor and then treated with the following liquid per sixty heavy hides:—1 cwt. ground bark, 1 cwt. ground valonia, 2 lbs. powdered zinc. The hides are placed in layers and the zinc powder thrown in between. After lying one or two weeks they are worked in the usual manner.

H. A. CLARK, Boston: *Recovering caoutchouc and gutta-percha from waste*. (Eng. P., No. 229, Jan. 18, 1881.)—Boiling with water to separate the greater part of the sulphur, and mixing with 2 to 10% of palm oil. The whole is then heated and exposed to the action of turpentine or camphene vapors until it becomes plastic. Finally 2 to 10% of resinous substances are added.

W. SMITH: *Solvent and detergent*. (Eng. P., No. 2025, May, 1880.)—Petroleum distillates of low boiling points are treated with chlorine gas until their disagreeable odor disappears. They are then mixed with milk of lime and soda lye and re-distilled.

H. KROPPF, Nordhausen: *Improvements in ammonia ice machines*. (Germ. P., No. 13853, Nov. 24, 1880.)—The inventor proposes to employ two or more ammonia vessels instead of one, and to condense the gas from them in a common receptacle, and also to evaporate it in a common ice generator.

R. C. ANDERSON, Woodgreen, Middlesex: *Galvanic battery*. (Eng. P., No. 2564, June 23, 1880.)—The inventor proposes to prevent the deposition of copper upon the positive element of a Daniell's battery, by employing an intermediate cell containing a liquid of high conductivity and a metal, such as iron, capable of precipitating the copper.



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OCTOBER, 1881.

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July	6, 1876	Bailey, E. H., Bethlehem, Pa.
March	7, 1878	Baker, W. H., Freeport, Ill.
April	6, 1876	Balentine, Walter, Agricultural Department, Washington, D. C.
Oct.	5, 1876	Barcena, Mariano, Museo Nacional, City of Mexico, Mexico.
April	6, 1876	Battershall, J. P., 76 Varick st., N. Y.
Oct.	7, 1881	Bauer, Harry von, 12 Frankfort st., N. Y.
June	1, 1876	Baumgarten, A., St. Lawrence Sugar Refining Co., Montreal, Canada.
April	6, 1876	Baumgarten, F. M., 179 16th st., San Francisco, Cal.
Jan.	3, 1878	Beckwith, Leonard F., Courtlandt and Church sts., N. Y.
April	6, 1876	Behr, Arno, care of Matthiessen & Wiechers, Jersey City, N. J.
July	6, 1876	Belcher, G. W., St. Louis, Mo.
April	3, 1879	Benjamin, Marcus, 10 Barclay st., N. Y.
Oct.	7, 1880	Bihn, G. T., 523 Tasker st., Philadelphia, Pa.
Nov.	1, 1877	Blanpied, Benj., Dartmouth Agricultural Coll., Hanover, N. H.
April	6, 1876	*Blossom, Thomas M., San Francisco, Cal.
April	6, 1876	Bodley, Miss Rachel L., Woman's Medical College, Phila., Pa.
Dec.	7, 1876	Booth, J. C., United States Mint, Philadelphia, Pa.
April	6, 1876	Bourgougnon, A., 91 Duane st., N. Y.
March	1, 1880	Bragg, Everett B., 33 Park place, N. Y.
April	6, 1876	Brinley, Charles A., Midvale Steel Works, Germantown, Philadelphia, Pa.
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Sept.	7, 1876	Buck, C. Elton, Wilmington, Del.
April	6, 1876	*Cairns, F. A., N. Y.
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April	6, 1876	Casamajor, P., care Havemeyer & Elder, Brooklyn, E.D., N. Y.
April	6, 1876	† Chandler, Chas. F., School of Mines, Columbia College, N. Y.
Sept.	7, 1877	Chase, Alonzo, Room 123, Cochrane B'ld'g, Washington, D. C.
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Nov. 7, 1878	Cruse, E. F., 20 Bridge st., Brooklyn, N. Y.
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April 6, 1876	Doremus, Charles A., 127 E. 27th st., N. Y.
Dec. 2, 1879	Dosher, Henry, 142 Rose st., Brooklyn, E. D.
April 6, 1876	Douglass, Silas H., Ann Arbor, Mich.
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April 6, 1876	Ebert, A. E., Peoria Sugar Refinery, Peoria, Ill.
March 6, 1879	Eimer, August, 220 E. 19th st., N. Y.
April 6, 1876	Elliot, Arthur H., School of Mines, Columbia College, N. Y.
Oct. 5, 1876	Ellis, William H., Toronto, Canada.
April 6, 1876	Endemann, H., 33 Nassau st., N. Y.
April 6, 1876	Englehardt, Francis E., Syracuse Salt Co., Syracuse, N. Y.
March 6, 1879	Erhardt, C. F., care Chas. Pfizer & Co., 81 Maiden Lane, N. Y.
Dec. 5, 1878	Everhardt, Edgar, Stevens Institute, Hoboken, N. J.
April 6, 1876	Fesquet, Adolphe A., 1322 Marshall st., Philadelphia, Pa.
June 1, 1876	Fisher, R. A., 2239 St. Albans st., Philadelphia, Pa.
Dec. 2, 1879	Freish, H., 136 Church st., Bridenbarg, Philadelphia, Pa.
Dec. 2, 1880	Friedburg, Dr. Henry, 16 Bedford ave., Brooklyn, E. D., N. Y.
April 6, 1876	Fristoe, Edward E., Columbian College, Washington, D. C.
May 1, 1879	Gallatin, Albert H., Room 26, Cooper Union, N. Y.
April 6, 1876	Genth, Fred. A., University of Pennsylvania, Phila., Pa.
Feb. 11, 1881	Gerber, Dr. N., Amer. Cond. Milk Co., Little Falls, N. Y.
April 6, 1876	Gesner, G. W., 81 John st., N. Y.
March 1, 1877	Geyer, Wm. E., Stevens Institute, Hoboken, N. J.
Oct. 14, 1879	Giessecke, Dr., 19 W. Swan st., Buffalo, N. Y.
April 6, 1876	Gifford, W. E., 54 Pine st., N. Y.
April 6, 1876	Goessman, Charles A., Amherst, Mass.
April 6, 1876	*Goldmark, J., 13 Barclay st., N. Y.
Oct. 5, 1876	Goldschmidt, S. A., 59 Liberty st., N. Y.
April 6, 1876	Green, Trail, Easton, Pa.
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- April 6, 1876 Griffin, Roger B., care W. M. Habirshaw, 159 Front st., N. Y.  
 May 6, 1881 Grote, Dr. Otto, 59-61 Goerck st., N. Y.  
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 Dec. 2, 1880 Hale, Albert C., State School of Mines, Golden, Col.  
 June 3, 1881 Hallock, A. P., N. Y. Gaslight Co., ave. A and 21st st., N. Y.  
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 April 6, 1876 Hardin, M. B., Virginia Military Institute, Lexington, Va.  
 April 6, 1876 Harnish, H. H., 113 Water st., N. Y.  
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 April 6, 1876 Hasenclever, Maurice, 1 Appleton st., Lawrence, Mass.  
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 N. Y.
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 June 1, 1876 Henes, W. F., 1620 Third ave., N. Y.  
 April 6, 1876 Herreshoff, J. B. F., 135 Clinton st., Brooklyn, N. Y.  
 April 6, 1876 Heyx, Thomas, Toronto, Canada.  
 Oct. 7, 1881 Hiepe, H. C., 102 John st., N. Y.  
 April 6, 1876 Hill, Nathaniel P., Denver, Col.  
 Jan. 4, 1877 Hill, W. M., U. S. Torpedo Station, Newport, R. I.  
 April 6, 1876 Hills, William B., Cambridge, Mass.  
 March 1, 1877 Hindley, Robert C., Racine College, Racine, Wis.  
 April 6, 1876 Homer, Chas. S., care of Valentine & Co., 323 Pearl st., N. Y.  
 Feb. 11, 1881 Hopke, T. M., 17 Cedar st., N. Y.  
 April 1, 1881 Hoppock, A. E., 103 E. 148th st., N. Y.  
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- May 1, 1879 Ihlsing, M. C., Columbia College, N. Y.
- June 6, 1878 Jenks, A. M., care of Havemeyer, Eastwick & Co., Jersey  
 City, N. J.
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 July 6, 1876 Johnson, Samuel W., New Haven, Ct.
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 April 6, 1876 Kersting, Herman, 324 Princeton st., East Boston, Mass.  
 July 6, 1876 Koenig, G. A., University of Pennsylvania, Philadelphia, Pa.  
 April 6, 1876 Krackowitzer, S., 512 E. 17th st., N. Y.  
 April 6, 1876 Krause, O. H., care Matthiessen & Wiechers, Jersey City, N. J.
- Feb. 6, 1879 Lamothe, Charles D., P. O. Box 1567, N. Y.  
 April 6, 1876 Land, William J., Box 305, Atlanta, Ga.  
 Sept. 7, 1876 Langley, John W., Ann Arbor, Mich.

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Nov.	1, 1877	Lecompte, Charles, Tenafly, N. J. Gone to France.
April	6, 1876	Ledoux, A. R., 17 Cedar st., N. Y.
Jan.	8, 1878	Leeds, A. R., Stevens Institute, Hoboken, N. J.
Jan.	8, 1878	Lees, Wm. T., N. Y. Gaslight Co., ave. A and 31st st., N. Y.
April	6, 1876	Liebig, G. A., 87 Exchange place, Baltimore, Md.
July	6, 1876	Lillie, Samuel Morris, 333 Pine st., Philadelphia, Pa.
Oct.	7, 1881	Leman, W. P., 305 W. 55th st., N. Y.
April	6, 1876	Lindsley, J. Berrian, Nashville, Tenn.
April	6, 1876	Lipps, John S., 24 Park place, N. Y.
Dec.	2, 1880	Lungwitz, Theo., care of H. E. Niese, Jersey City, N. J.
April	6, 1876	Lupton, N. T., Vanderbilt University, Nashville, Tenn.
May	2, 1878	Luthy, D., 220 Church st., Philadelphia, Pa.
April	6, 1876	McIntire, Chas. M., Jr., 311 N. Hampton st., Easton, Pa.
Jan.	2, 1879	*McIntire, Henry M., 1136 Washington st., Easton, Pa.
June	6, 1878	MacIntosh, J. B., 55 Garden st., Hoboken, N. J.
April	6, 1876	Maish, John M., College of Pharmacy, Philadelphia, Pa.
April	6, 1876	Mallet, J. W., University of Virginia, Albemarle Co., Va.
Feb.	6, 1879	Marchand, Charles, P. O. Box 1567, N. Y.
April	6, 1876	Martin, Wm. J., Davidson College, Mecklenburg Co., N. C.
Sept.	7, 1876	Martins, R. A., care of Actien-Gesellschaft für Anilin Fabrikation, Berlin, Germany.
May	6, 1878	Mattison, J. G., Hastings-on-Hudson, N. Y.
Feb.	1, 1877	Means, Alexander, Oxford, Newton Co., Ga.
April	6, 1876	*Merrick, John M., Boston, Mass.
April	6, 1876	Mew, Wm. M., 422 5th st., Washington, D. C.
Dec.	1, 1879	Meyers, Henry C., 46 Cliff st., N. Y.
June	1, 1876	Miller, George M., 20 Broad st., N. Y.
April	6, 1876	Moore, Gideon E., 69 Liberty st., N. Y.
April	6, 1876	Morrill, Thomas T., Cambria Iron Works, Johnstown, Pa.
April	6, 1876	Morton, Henry, Stevens Institute, Hoboken, N. J.
June	1, 1871	Mott, H. A., Jr., 105 Water st., N. Y.
April	6, 1876	Munroe, Charles E., U. S. Naval Academy, Annapolis, Md.
March	7, 1878	Munroe, H. S., School of Mines, Columbia College, N. Y.
April	6, 1876	Nason, Henry B., Troy, N. Y.
April	6, 1876	Niese, H. E., care of Matthiessen & Wiechers, Jersey City, N. J.
April	6, 1876	Nichols, W. H., 41 Cedar st., N. Y.
May	3, 1877	Norton, Thomas, 2 Route Landai, Saint Denis, Paris, France.
Dec.	2, 1880	O'Connor, J. D., School of Mines, Columbia College, N. Y.
Nov.	19, 1879	Oothout, William, Bellevue Hospital, N. Y.
Dec.	6, 1877	Orth, A., care of F. Bredt & Co., N. Y.
July	6, 1876	Parker, T. J., Box 69, Bergen Point, N. J.
April	6, 1876	Parsons, C. C., 542 E. 20th st., N. Y.

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July 6, 1876	Pearce, Richard, Denver, Col.
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April 6, 1876	Perkins, Maurice, Schenectady, N. Y.
April 6, 1876	Phelps, George N., 127 First place, Brooklyn, N. Y.
July 11, 1878	Phillips, William B., University of North Carolina, Chapel Hill, N. C.
Nov. 2, 1876	Pickhardt, Carl W., Pickhardt & Kuttroff, cor. of Liberty and New Church sts., N. Y.
Dec. 2, 1880	Pigeon, Narcise, 128th st. and 4th ave., N. Y.
April 6, 1876	Pirz, Anthony, Long Island City, N. Y.
Oct. 5, 1876	Pitkin, Charles A., Box 52, Braintree, Mass.
March 6, 1879	Pool, Franklin V., 191 Roseville ave., Newark, N. J.
Oct. 7, 1880	Pool, Herman, Buffalo, N. Y.
Feb. 6, 1879	Potter, T. W., 24 Cliff st., N. Y.
April 6, 1876	Prescott, Albert B., Ann Arbor, Mich.
March 1, 1877	Prochazka, George A., 162 2d ave., N. Y.
Oct. 7, 1881	Radenhausen, P., Stevens Institute, Hoboken, N. J.
April 6, 1876	Rice, Charles, Bellevue Hospital, N. Y.
April 6, 1876	Ricketts, P. de P., School of Mines, Columbia College, N. Y.
April 6, 1876	Rising, William B., University of California, Oakland, Cal.
June 1, 1876	Robbins, Charles A., 91 Fulton st., N. Y.
March 4, 1878	Roberts, W. Chandler, Royal Mint, London, Eng.
March 4, 1878	Rogers, F. M., 95 Liberty st., N. Y.
April 6, 1876	Rogers, Robert E., Jefferson Medical College, Phila., Pa.
Nov. 1, 1877	Rossi, Auguste J., 22 W. 11th st., N. Y.
July 11, 1878	Rupp, William, 961 3d ave., N. Y.
April 6, 1876	*St. John, Samuel, N. Y.
June 5, 1879	Schanck, J. Stillwell, Princeton College, Princeton, N. J.
Dec. 2, 1880	Schmich, Charles W., Rock Hill Iron and Coal Co., Orbisonia, Huntington Co., Pa.
Sept. 7, 1876	Schrage, William, Sheboygan, Wis.
April 6, 1876	Schultz, Charles H., cor. 17th st. and Broadway, N. Y.
April 6, 1876	Schweitzer, Paul, Columbia, Boone Co., Mo.
Dec. 2, 1880	Seaman, Henry J., Crane Iron Co., Catasauqua, Pa.
July 6, 1876	Seeley, Henry Martyn, Middlebury, Vt.
Jan. 1, 1880	Semper, C., Gray's Ferry Chem. Works, Philadelphia, Pa.
April 6, 1876	Shapleigh, Waldron, Freeport, Ill.
April 6, 1876	Sharpless, S. P., 114 State st., Boston, Mass.
Oct. 3, 1878	Shepard, Charles U., Jr., 20 Broad st., Charleston, S. C.
April 6, 1876	Sherer, E., 122 Front st., N. Y.
Feb. 7, 1879	Sherer, J. A., 122 Front st., N. Y.
April 6, 1876	Silliman, Benjamin, New Haven, Ct.
April 6, 1876	Simon, Wm., Maryland College of Pharmacy, Baltimore, Md.
Feb. 11, 1881	Slade, James F., 5 E. 38th st., N. Y.
May 3, 1877	Sloan, T. O'C., 55 Pine st., N. Y.
Nov. 1, 1877	Smith, Hanbury, 35 Union square, N. Y.
March 1, 1877	Squibb, E. R., 36 Doughty st., Brooklyn, N. Y.



## DATE OF ELECTION.

Oct.	7, 1880	Stahl, Dr. Karl, N. W. Fertz. Co., Union Stock Yards, Chicago, Ill.
June	5, 1879	Stebbins, James H., Jr., 20 E. 37th st., N. Y.
April	6, 1876	Sticht, John C., care of Charles Pfizer & Co., 81 Maiden Lane, N. Y.
March	6, 1879	Stillman, T. B., 40 Broadway, N. Y.
April	6, 1876	Stillwell, Charles M., 55 Fulton st., N. Y.
Dec.	6, 1877	Striedinger, J. H., 17 University place, N. Y. Gone to S. Amer.
July	6, 1876	Sweeny, William S., Easton, Pa.
April	6, 1876	Taylor, William H., 606 E. Grace st., Richmond, Va.
Oct.	5, 1876	Terne, Bruno, 1628 S. 5th st., Philadelphia, Pa.
Feb.	7, 1878	Thompson, A. L., 52 Sharp st., Baltimore, Md.
Jan.	3, 1879	Thompson, M. S., 58 Wall st., N. Y.
April	6, 1876	Tilden, William C., 127 Hudson st., N. Y.
May	1, 1881	Tonnellé, Theodore, Wampum Cement and Lime Co., Newcastle, Lawrence Co., Pa.
Oct.	7, 1880	Torry, C. H., School of Mines, N. Y.
Feb.	7, 1878	Tscherniac, J., 118 Boulevard Maillot, Neuilly, Paris, France.
May	6, 1879	Tucker, J. H., care of McKean, Newhall & Borie, Philadelphia, Pa.
April	3, 1879	Van Slooten, William, 42 S. Derbigny st., New Orleans, La.
April	6, 1876	Vaughn, Henry W., Providence, R. I.
Feb.	11, 1881	Venable, T. S., Chapel Hill, N. C.
Dec.	6, 1877	Wait, Charles E., Missouri School of Mines, Rolla, Mo.
Jan.	3, 1878	Waldstein, Martin E., 92 New Church st., N. Y.
April	6, 1876	Waller, Elwyn, School of Mines, Columbia College, N. Y.
April	6, 1876	*Walz, Isidor, N. Y.
June	6, 1878	Ware, Louis S., St. George Hotel, Philadelphia, Pa.
April	6, 1876	Weber, Henry A., Ill. Industrial University, Champaigne, Ill.
April	6, 1876	Wendt, Charles J., care of Charles Pfizer & Co., 81 Maiden Lane, N. Y.
Sept.	7, 1876	Wenzell, Wm. T., 852 Market st., San Francisco, Cal.
March	6, 1879	Wheeler, C. G., University of Chicago, Chicago, Ill.
Dec.	2, 1880	Wiegman, F. G.
Jan.	2, 1879	Wigner, G. W., 79 Great Tower st., London, England.
Dec.	1, 1879	Williams, E. H., 101 N. 32d st., Philadelphia, Pa.
April	6, 1876	Williams, Charles P., Rolla, Mo.
June	5, 1879	Wilson, M. C., Waco, Texas.
April	6, 1876	Winans, Horatio N., 1551 Broadway, N. Y.
Dec.	2, 1880	Wittman, Byron N., Pittsburg Steel Casting Co., Pittsburg, Pa.
July	6, 1876	Wolf, Theo. R., Delaware College, Newark, Del.
April	6, 1876	Wormley, Theo. G., University of Pennsylvania, Phila., Pa.
July	6, 1876	Youmans, W. J., care of D. Appleton & Co., 1 Bond st., N. Y.
July	6, 1876	Youmans, E. L., care of D. Appleton & Co., 1 Bond st., N. Y.

## ASSOCIATE MEMBERS.

---

**DATE OF ELECTION.**

June	1, 1876	Adler, J., 744 Lexington ave., N. Y.
June	1, 1876	Bartlett, Willard, 110 Broadway, N. Y.
March	6, 1879	Binns, Joseph, cor. Pearl and Cedar sts., N. Y.
March	4, 1880	Booraem, J. V. V., N. 3d and 1st sts., Brooklyn, E. D., N. Y.
June	6, 1878	Bower, Henry, Gray's Ferry Road, Philadelphia, Pa.
June	3, 1880	Cochrane, A., 55 Kilby st., Boston, Mass.
June	3, 1880	Cochrane, Hugh, 55 Kilby st., Boston, Mass.
July	6, 1876	Day, Austin G., 120 Broadway, N. Y.
June	1, 1876	Day, Walter De F., 301 Mott st., N. Y.
March	6, 1879	Despard, A. W., 20 Liberty st., N. Y.
July	6, 1876	Dickerson, Edward N., Staats-Zeitung Building, N. Y.
April	5, 1877	Donner, J. O., care of Decastro & Donner, foot of South 9th st., Brooklyn, N. Y.
April	5, 1877	Donner, Oscar W., care of Decastro & Donner, foot of North 3rd st., Brooklyn, N. Y.
Feb.	11, 1881	Dunham, E. K., School of Mines, N. Y.
Jan.	4, 1877	Elder, Fred H., care of Havemeyer & Elder, 117 Wall st., N. Y.
July	6, 1876	Elder, George, Jr., 19 West 48th st., N. Y.
Oct.	5, 1876	Elmenhorst, W. R., St. Lawrence Sugar Refining Co., Montreal, Canada.
July	6, 1876	Gandolfo, Joseph, 41 Cedar st., N. Y.
April	4, 1878	Harding, G., 237 Broadway, N. Y.
Sept.	7, 1876	Havemeyer, C. W., care of Havemeyer Bros., Brooklyn, E. D., N. Y.
April	5, 1877	Havemeyer, Henry, 89 Wall st., N. Y.
July	6, 1876	Havemeyer, Henry O., 117 Wall st., N. Y.
July	6, 1876	Havemeyer, Theodore A., 117 Wall st., N. Y.
April	5, 1877	Heller, Fred., 55 Maiden Lane, N. Y.
June	1, 1876	Hinds, Joseph, care of Havemeyer & Elder, Brooklyn, E. D., N. Y.
Dec.	6, 1876	Holly, John J., 63 Beaver st., N. Y.
June	1, 1876	Holt, Henry, 12 East 23d st., N. Y.
April	5, 1877	Kalbfleisch, A. M., cor. Fulton and Cliff sts., N. Y.
April	5, 1877	Kalbfleisch, Franklin A., cor. Fulton and Cliff sts., N. Y.
Nov.	7, 1876	Kuttroff, Adolph, Wm. Pickhardt & Kuttroff, cor. New Church and Liberty sts., N. Y.
Dec.	2, 1880	Kyte, Charles, Jr., 3 East India ave., London, E. C.

## DATE OF ELECTION.

Feb. 6, 1879	Leggett, E. W., Elizabethport, N. J.
March 1, 1877	Lennig, Charles, Philadelphia, Pa.
July 6, 1876	Mapes, Charles V., 158 Front st., N. Y.
July 6, 1876	Matthiessen, F. O., 106 Wall st., N. Y.
Nov. 2, 1876	Merrill, Rufus L., care of Downer Kerosene Oil Co., Boston, Mass.
April 5, 1877	Merz, Henry, care of Heller & Merz, 55 Maiden Lane, N. Y.
July 6, 1877	Miles, William A., 57 Christie st., N. Y.
June 6, 1876	Moran, Charles, 12 E. 53d st., N. Y.
April 5, 1877	Nichols, G. H., 41 Cedar st., N. Y.
Sept. 7, 1876	Page, George Shepard, 49 Wall st., N. Y.
July 6, 1876	Parsons, W. J., 66 Water st., Brooklyn, N. Y.,
July 6, 1876	Plympton, G. M., 245 Broadway, N. Y.
Nov. 2, 1876	Pickhardt, William, Wm. Pickhardt & Kuttroff, cor. New Church and Liberty sts., N. Y.
March 7, 1878	Prentice, W. P., 301 Mott st., N. Y.
May 3, 1877	Renwick, Edward S., 16 Murray st., N. Y.
Sept. 7, 1876	*Russell, W. D., Newark, N. J.
Oct. 7, 1880	Savage, J. R., Frankfort Chemical Works, Philadelphia, Pa.
May 6, 1881	Sawyer, C. P., High Point, Guilford Co., N. C.
Oct. 5, 1876	Senff, Charles H., care of Havemeyer & Elder, 117 Wall st., N. Y.
July 6, 1876	Senff, Frederick W., 558 Madison ave., N. Y.
April 5, 1877	Schroeder, E., 100 Glenwood ave., Jersey City, N. J.
April 5, 1877	Sieghoertner, A. L., Belcher Sugar Refining Co., St. Louis, Mo.
Jan. 4, 1877	Squier, Stewart C., 113 Liberty st., N. Y.
Sept. 7, 1876	Stoddard, Wm. O., 178 Greenwich st., N. Y.
April 5, 1877	Stursberg, Julius A., Brooklyn Sugar Refinery, foot of South 9th st., Brooklyn, N. Y.
May 6, 1881	Van Sinderen, A. Howard, School of Mines, N. Y.
Dec. 2, 1880	Vulte, Herman T., School of Mines, N. Y.
May 2, 1878	Weston, E. D., 135 Lake st., Chicago, Ill.
May 2, 1878	Williamson, D. D., 683 Madison ave., N. Y.
Feb. 6, 1879	Wingate, C. F., 140 William st., N. Y.
March 6, 1879	Ziegler, William, care of Royal Baking Powder Co., 91 Duane st., N. Y.

# JOURNAL

OF THE

## ERRATA.

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**Pages 49, 57, 62, and 72, read Hammer in place of Hammel all through,**

**Page 49, 4th line from top, read Zinc for Indigo.**

“ 49, 14	“	“	botom,	“	Employed for Examined.
“ 49, 8	“	“	“	“	Gallic acid for Tannic acid.
“ 49, 7	“	“	“	“	which for it.
“ 52, 13	“	“	“	“	Compensated for complicated.
“ 53, 14	“	“	top,	“	fine for five.
“ 55, 8	“	“	“	“	Tan for air.
“ 56, 14	“	“	bottom,	“	Insert the word Barkometer before the second word in the line.
“ 56, 10	“	“	“	“	Filings for Filtering.

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1882.

JOURNAL  
OF THE  
AMERICAN CHEMICAL SOCIETY.

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VOLUME IV.

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*PUBLICATION COMMITTEE*

ELWYN WALLER,  
CHARLES A. DOREMUS,  
L. H. FRIEDBURG.

*ABSTRACTORS*

A. H. ELLIOTT,	L. H. FRIEDBURG,
A. BOURGOUGNON,	O. H. KRAUSE,
PERCY NEYMANN.	

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# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

---

*Meeting held January 6th, 1882.*

The meeting was called to order at 8:20 p. m., Dr. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

The report of the board of directors was read and approved.

After which Mr. Elliott stated that he attended Mr. John Lipps' funeral together with Dr. Waller and some other gentlemen, and that the expenses incurred amounted to about \$116, of which he had only raised \$96.50 by subscription, the balance having been paid out of his own pocket.

The recording secretary then moved that Mr. Elliott be reimbursed from the society's funds.

It was then amended, that Mr. Elliott's claim be brought up at the next meeting of the board of directors for consideration.

This on motion was carried.

After which the following gentlemen were duly elected members of the society:

Clement Le Boutillier, associate member, proposed by A. H. Elliott, T. O'C. Sloane, A. R. Leeds.

Victor M. Braschi, regular member, proposed by A. H. Elliott, T. O'C. Sloane, A. R. Leeds.

F. G. Wiechman, regular member, proposed by A. H. Elliott, T. O'C. Sloane, A. R. Leeds.

Lucius Pitkin, regular member, proposed by A. H. Elliott, T. O'C. Sloane, A. R. Leeds.

Charles A. Wittmack, associate member, proposed by A. H. Elliott, T. O'C. Sloane, A. R. Leeds.

Dr. Henry Rau, regular member, proposed by James H. Stebbins, Jr., O. M. Jenks, A. R. Leeds.

Dr. J. A. Peters, regular member, proposed by S. A. Goldschmidt, C. C. Parsons, James H. Stebbins, Jr.

Dr. J. Rudesch, regular member, proposed by M. Alsberg, James H. Stebbins, Jr., H. Endemann.

After which Mr. Orazio Lugo was nominated as a new member.

Dr. Endemann then stated, in behalf of the committee on papers

and publications, that he did not expect the next number of the Journal for 1881 would be out before February 1st, 1882.

After which the first paper of the evening, "Upon the influence of light, upon certain metallic oxides," by Dr. A. R. Leeds, was read.

After some questions by Mr. Elliott, the second paper, "On the estimation of tannic acid, in tanning materials," by Mr. Nelson H. Darton, followed.

This paper caused a very lively discussion, between some of the gentlemen present.

After which the third paper, "On a convenient and accurate form for a weighing flask, to deliver as a burette," by Mr. Nelson H. Darton, was read.

After some remarks by Dr. Grothe and others, Mr. Elliott announced the death of Dr. John W. Draper, and moved that a committee of two be appointed, to wait on Prof. Chandler, and request him to draw up an obituary notice. This being seconded was duly carried.

Dr. Friedburg then described a new test paper, manufactured by Toch, Grothe & Co., which has the property of distinguishing between mineral and organic acids.

This paper has a blue shade, and is not acted upon by organic acids.

Dr. Friedburg then proposed that our *Conversazioni* should be made more interesting, and urged that some paper or theme should be settled upon beforehand, the title of which should be sent to the recording secretary, to be announced on the postal cards.

This being seconded, was duly carried.

After which the recording secretary stated that he would give a paper on artificial indigo, with samples, for the next *Conversazione*.

Mr. Casamajor then announced the resignation of Mr. W. B. Hills and Chas. F. Wingate, both of which were duly accepted.

Mr. Elliott then asked whether any action had been taken about members that are in arrears of dues.

This being answered in the negative, Mr. Casamajor then moved that all members in arrears of dues up to Dec. 31, 1880, be dropped from the rolls.

This being seconded, was carried unanimously. After which the meeting adjourned.

JAMES H. STEBBINS, JR.,  
Recording Secretary.

## I. UPON THE REDUCTION OF METALLIC OXIDES IN SUNLIGHT.

BY DR. ALBERT R. LEEDS.

During the course of some experiments upon the measurement of solar actinism, I was led to subject certain metallic oxides to the influence of sun-light during a very prolonged period and under somewhat novel conditions. The oxides were contained in W-shaped tubes, from which the air was displaced by a current of pure and dry hydrogen continued during five hours. After exhausting the atmosphere of hydrogen as completely as possible, the tubes were sealed. The oxides were placed in one bend of the tube, and in the other bend metallic sodium, or, in some experiments, anhydrous cupric sulphate. All of the tubes were filled upon the 10th of November, 1880, and exposed at a southern window until Nov. 10th, 1881, when they were examined and opened.

I. A tube with 5.2256 grms. mercurous oxide in one bend, and sodium in the other. The oxide was largely reduced to metallic mercury, which formed a mirror with globules, and to *yellow* mercuric oxide. The sodium was covered with a white crust. On account of the mingling of the globules of mercury with the reduced and unreduced mercurous oxide, no quantitative estimations could be effected.

II. A V-tube containing 5.4064 grms. mercurous oxide, but no sodium. A mercury mirror was formed, and the residue consisted of a mixture of mercurous oxide and *yellow* mercuric oxide. As in the preceding experiment no red oxide of mercury was found.

The two preceding experiments are at variance with the results of Dulk, quoted in Gmelin-Krauts Handb. der Anorg. Chemic. Vol. I, p. 825, according to which the mercurous oxide was resolved into mercury and *red* oxide of mercury.

III. A W-tube containing 6.3597 grms. red oxide of mercury in one bend and metallic sodium in the other. No decomposition ensued, the weight remaining unaltered. This result likewise differs from that of Dulk (loc. cit.), who found that dry mercuric oxide lost in 4 months under colorless glass 0.9, under violet 0.5, under green 0.2 and under red 0.1 per cent. of oxygen. At the same time its color changed to gray, most strikingly under colorless, but scarcely perceptibly under red glass.

IV. A W-tube containing 3.043 grms. of pure  $\text{PbO}_2$  in one bend

and metallic sodium in the other. The weight and appearance of the peroxide remained unaltered.

This result differs from that of Dulk (*loc. cit.*), the brown oxide of lead being decomposed into oxygen and the red oxide.

V. 4.057 grms. of  $\text{PbO}_2$  was used in this experiment, but instead of sodium, anhydrous cupric sulphate was placed in the other bend of the tube. Neither the lead peroxide nor the copper salt changed in appearance, nor the former in weight.

---

## II. ON THE ESTIMATION OF TANNIC ACID IN TANNING MATERIALS, ETC.

By NELSON H. DARTON.

As the exact determination of the proportions of tannic acid is a problem of comparatively difficult solution many methods have been proposed to facilitate this, but, as a rule, have yielded very discordant results when compared with the actual weights of the leather formed from it. Thus tanners have lost all faith in chemists generally. Some prominent tanners have informed me that they have sent spent barks containing less than one per cent of tannic acid to chemists in this city and they have returned results of from 7 per cent. to 4 per cent. These results were generally obtained by Hammels hide method or that of Lœwenthal, as modified by Neubauer, or even by the old gelatin method, which is now seldom used.

In my intimate connections with the tanning trade I seriously felt the defects in and want of a proper method, and not being able to find one of sufficient accuracy sought out one that would answer the conditions of speed and accuracy, and this I will detail below, merely mentioning that in numerous and exhaustive trials I have found it to yield results as near as within  $\frac{1}{4}$  per cent of the actual value. Lœwenthal's modification of his own method is not nearly as accurate as this, as I will show in a subsequent paper.

The bark, etc., is taken dry and finely cut, powdered or crushed. 20 grammes of this is weighed out, placed in a flask with sufficient water and the tannic acid thoroughly exhausted by repeated treatments with boiling water. The filtrate is mixed with about 25 cc. of dilute sulphuric acid and again filtered after making the bulk up to one litre. Standard solutions are then prepared. The first of tannic acid containing two grammes to a litre. The pure tannic acid must be used. 2nd, a solution contain-

ing to a litre 1.5 grammes of permanganate of potassium, 3rd, a solution of indigo carmine of such a strength that it must require twice as much of the permanganate solution as an equal bulk of the tannin solution, that is about 3 grammes to a litre. 4th, a 10 per cent solution of ammonio-sulphate of copper in ammoniacal water. 5th, a dilute sulphuric acid, one of acid to five of water. A convenient measure of the indigo is taken, say 20cc. It is diluted to about  $\frac{3}{4}$  of a litre, acidulated with the acid and titrated with the permanganate. Another amount of the indigo and the same of tannic acid solution are taken and titrated as before. This is then repeated, using the decoction of the bark. About 60 cc. of the last is then taken and about the same of the copper solution, these are mixed together, some ammonia solution added and the mixture filtered from the tannate of copper which precipitates. An equivalent amount of this filtrate is titrated as before and from these data the amount of tannic acid calculated by the following formula.

The amount of permanganate used for the indigo and tannin together, less that used by the indigo, is to the permanganate used for the decoction, less that necessary for the indigo present and an equivalent amount of the filtrate noted, as the amount of tannic acid present in the litre of standard solution, is to the quantity of tannic acid in 20 grammes of the bark (a litre of decoction). The process may be executed in about one half hour after the decoction is obtained, but as eight or ten of the determinations may be carried on at once without loss of time it is by far the shortest method known.

This process has been in constant use in my laboratory for some time back, and as I have much of this work constantly to attend to I have thoroughly tested its value and am assured the main error is not over  $\frac{1}{4}$  per cent. as I will show in my subsequent paper.

This method, somewhat extended, is applicable to the determination of the gallic acid at the same time. It is simply in treating a portion of the filtrate from the tannate of copper with a solution of bichloride of mercury, and after filtering this off and separating the excess of Hg, titrating the filtrate, using the calculations similar to the above, the amount of gallic acid may be readily arrived at. The relation of the permanganate to a gallic acid solution for the first member of the equation should be previously ascertained, although it differs but slightly from the tannic acid.

I then proceeded to investigate the purity and properties of the tannate of copper upon which the whole of the method depends. Two tannates of copper were prepared, one from the pure

tannic acid, the other from the decoction of hemlock bark and the two were compared, the first, from the pure tannic acid, contained 27.72 per cent. of copper oxide and the other 27.58 per cent., within 99.5 per cent. of each other. By combustion they both yielded essentially the same formula. In both cases they were in black lustrous masses quite insoluble in cold but partly soluble in hot or boiling water, from which it is deposited on cooling. In alcohol, gallic acid solutions and in presence of alkalies they are very insoluble but quite soluble in diluted acids.

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### III. ON A CONVENIENT AND ACCURATE FORM OF WEIGHING FLASK TO DELIVER AS A BURETTE.

BY NELSON H. DARTON.

In many volumetric processes, the end reaction is so sharp that the small amount of solution required to produce it is too small to be perceptible in the reading of an ordinary burette. Readings to 0.05 cc. are as close as can be usually seen. Differences of temperature, which affect both the bulk of the solution and the capacity of the burette, are also sources of error which must be avoided if we wish to attain great precision.

In cases where extreme accuracy is required, the method of weighing, instead of measuring the amount of solution used, is to be preferred.

Two or three forms of apparatus for this purpose have been proposed, but they all are open to two objections. 1st. That the delivery tube is too large to deliver less than 0.05 cc., or if fine enough to deliver it, a sufficient amount cannot be delivered conveniently at the start to bring the solution upon which one is working near to the point of change, and 2d. that regulating the admission of the air to the apparatus distracts the operator's attention at the critical moment.

I have constructed and used an apparatus which is not open to the above objections. Two forms are used. The first for liquids which may suffer an alteration of standard by contact with organic substance, as cork or rubber, consists of a flask with a Gay-Lussac delivery tube with a fine delivery jet. The stopper is perforated for a glass tube, connecting by a short rubber tube with another tube drawn to a fine point to regulate the admission of air to the flask. A small pinch cock on the rubber tube completes the control of the air supply, and consequently of the delivery from the flask. With the

apparatus I use three or more of these tubes for regulating the air supply, which can be weighed with the flask and changed as required.

The second form of apparatus is essentially the same, except that the solution is delivered from the flask by a small siphon passing through the cork, the supply being regulated as before.

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## OBITUARY.

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### JOSEPH GOLDMARK.

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From the small band of men by whom the project of an American Chemical Society was agitated, we have lost one who entered into it with enthusiasm and who, ever since the founding of the society, has rejoiced in its existence—Dr. Joseph Goldmark.

Although born and educated abroad and strongly attached to the land of his birth, the active part of Dr. Goldmark's life was passed in the city of New York, where he built up his large manufacturing business and where all his interests were centered.

He was born in the small town of Kreuz in Hungary.

At the age of seventeen he entered the University of Vienna, where he took his degree of Doctor of Medicine. At the same time he devoted himself to the study of chemistry with great zeal, working in the laboratory under Prof. Schroetter. Here he applied himself to the investigation of amorphous phosphorus, many of the remarkable properties of which he claims to have discovered. It was his intention to assert his claim to these discoveries in the scientific journals, and on the occasion of his last visit to Europe he was engaged in collecting material to establish his title to them.

In the revolution of 1848, which brought the first dawn of political liberty to Austria, Joseph Goldmark took part as a leader. In connection with such men as Fischhof, Violand, Fuster and others he will be remembered as the bold and enthusiastic advocate of reform.

In the stormy days of March, 1848 he was at the head of the Legion of Students who left their lecture hall, opposed the professors, and fifteen hundred strong took possession of the Chamber of Deputies and forced the "Staende" to petition the emperor for



reforms, including liberty of the press, religious liberty, representation by the people, etc.

He was chosen as one of the twelve delegates sent by the students to the imperial palace, and took part in all the stirring measures of that memorable period.

But the glorious early days of the movement, so full of high aspirations and of enthusiasm for liberty, were followed by darker days, when the spirit of loyalty and moderation was supplanted by the blind fury of the mob.

It was in such days that the names of the exalted leaders became connected with the deeds of violence which they themselves held in abhorrence.

Thus when Latour, the hated Minister of War, fell (Oct. 6, 1848) slain by the populace, Goldmark and Fischhof were the two delegates who sought to save him, endangering their own lives to save the minister's.

When the brief reign of the multitude was over, and absolutism again set in, the leaders of the revolution were held to account for the wild deeds of the mob, and an unjust retribution fell upon innocent men.

Fischhof lingered long in prison, while Goldmark escaped across the border. But, indicted as the instigators of the murder of Latour, they were condemned to death on the testimony of one witness and the sentence was carried out in effigy.

Goldmark and other fugitives reached Switzerland in safety and thence proceeded to the United States in 1849.

After one year's practice of his profession, Dr. Goldmark abandoned it for the more congenial pursuit of experiment and manufacture. For some time he was engaged in experiments with fulminating powders, the manufacture of which he reduced to a comparatively safe process, and in 1858 he established in Brooklyn a factory for cartridges and percussion caps.

To this work he gave the best years of his life, personally superintending the manufacture, inventing machinery, patenting improvements and competing with foreign manufacturers, his chemical knowledge being seconded by considerable mechanical ingenuity.

In 1868 he returned once more to his native Austria, not to avail himself of the amnesty which the emperor had extended to the banished revolutionists, but in order to secure a just trial and to prove his innocence.



He returned—on the statute books still the condemned murderer—but, a trial being granted, he was triumphantly acquitted, overwhelming proof showing that the witness whose testimony had convicted him had been bribed to swear to false statements. And not only was the innocence of Dr. Goldmark thus fully demonstrated, but it was also made apparent in what regard the people still held the men of '48; for at the close of the trial a public ovation was tendered to him, in which all the students and professors of the university joined as well as many eminent men in public stations.

Returning to his adopted country Dr. Goldmark continued his quiet life as a private citizen, finding leisure in spite of numerous business cares for constant attention to the interests of the political situation as well as for scientific pursuits. Although he never held an office he devoted much of his time and means to the service of political reform, holding it to be the duty of every true man to give a part of his attention to public matters, discouraging though the field of party strife may be.

The responsibilities and cares connected with the management of his large business left him no time in the latter years of his life for the prosecution of original research in the domain of pure chemistry; but the part he took in the discussions at the meetings of the society and his conversation showed how well he had kept pace with the progress of science.

Repeatedly elected a member of the Board of Directors and chosen to serve on various committees, Dr. Goldmark devoted much of his time to the business of the society, giving it the benefit of his wide experience and sound judgment.

Upright, just, accomplished and of a genial disposition he was loved by those who enjoyed his friendship and respected by all who knew him. By his death the society loses a distinguished member and the community an exemplary citizen. He belonged to that class of men whose watchword is: "the best and highest only," and his lifework was thorough, reliable and self-forgetful.

He died at the age of sixty-two on April 18th 1881.

O. H. KRAUSE.

# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Room 1, University Building, February 3, 1882.*

The meeting was called to order at 8.20 P. M., Prof. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

The report of the Board of Directors was then read.

The Librarian then stated that he had received the missing numbers of the *Berichte der D. Chem. Gesell.* along with a number of other pamphlets, etc.

The Librarian on being asked whether he had taken any steps in regard to the subject brought up at the last meeting of the Board of Directors, stated that he had had a number of cards printed which he would furnish on application to any member desiring to use the library.

He furthermore stated that the janitor had consented to take down the names of all gentlemen desirous of using the library.

Dr. Geyer then stated that the \$19.50 due to Mr. Elliott for expenses incurred on Dr. J. Lipps' funeral had been collected and paid.

The Chairman of the Committee on Papers and Publications had no report.

The question of dropping members in arrears of dues was then brought up, and ended in the following motions by Mr. Casamajor.

1. That all members in arrears of dues to Dec. 31, 1879, be dropped from the rolls.

This, on being seconded, was carried unanimously.

2. That a committee, consisting of the Treasurer and the two Secretaries, be appointed to draw up a list of members whose dues have been properly paid. Carried.

Mr. Elliott then made some remarks about the publishing of the next number of the Society's Journal, and finally stated that at the next meeting of the Society, he would move an amendment to the Constitution relating to the Committee on Papers and Publications which he wishes to be increased to five.

Mr. Orazio Lugo was then elected as a regular member.

After which, Dr. Emil Hirshberg was nominated as a new member.

The first paper of the evening "On Crystallized Anhydrous Grape Sugar," by Dr. Arno Behr, was then read.

Some remarks by Dr. Waller then followed, after which the second paper "On the precipitation of tannic acid as tannate of copper," by Mr. Nelson H. Darton, was read.

After some remarks and questions by Dr. Grothe the third paper, "On the water supply of N. Y. City," by Dr. E. Waller, was read.

This paper provoked a lively discussion between Drs. Endemann & Waller.

Dr. Endemann then read a paper by title "On Heptane," by F. P. Venable, Ph. D.

After which the meeting adjourned.

JAMES H. STEBBINS, JR.,  
*Recording Secretary.*

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#### IV. ON CRYSTALLIZED ANHYDROUS GRAPE SUGAR.

DR. ARNO BEHR.

The general physical and chemical properties of crystallized anhydrous grape sugar are pretty well known, and I am only able in a few points to supplement this knowledge on this occasion. Anhydrous grape sugar in a state of purity has so far only been obtained from an alcoholic solution. Two years ago F. Soxhlet found that the best solvent for it is methylic alcohol, from which much larger and better developed crystals can be obtained than from the solution in ethylic alcohol. I have found that it can, even more easily, be prepared from a watery solution.

The principle that a crystal introduced into the supersaturated solution of the same substance induces crystallization, has long been applied to the practice of grape sugar manufacture. In order to hasten the hardening of the sugar a certain quantity of the already hardened sugar of a previous operation is stirred into the mass. But as the ordinary commercial grape sugar always contains the hydrate the crystallization so obtained is also that of the hydrate. I put the question to myself, what would happen if, instead of the hydrate, I introduced the anhydrous sugar into a concentrated solution of ordinary grape sugar. I tried the experiment and must confess that I had not much hope that anything else but crystallized hydrate would be the result, for I expected to see the anhydride transformed into the hydrate within the watery solution. I was agreeably surprised when, on the next morning, I found the glass filled with a neat crystallization of anhydrous grape sugar,

from which the liquid part could be easily drained. The few crystals of anhydride, far from being transformed into the hydrate, had induced an ample crystallization of their kind. The explanation of this fact is found in the following : In its crystalline form anhydrous grape sugar is not deliquescent even in very moist weather, and it is stable in comparatively dilute solutions of grape sugar. I have kept crystals exposed to the atmosphere of the laboratory through months and during moist weather without seeing them lose their sharp outlines and bright appearance, and I have repeatedly found the syrup drained from a crystallization of anhydrous sugar to contain as much as 26% of water.

The limits of concentration within which this crystallization can be obtained are rather wide, but in order to secure a good result the solution ought to contain from 12 to 13% of water. It is well not to allow the mass to cool rapidly or the temperature to fall much below 30° C. For, at a lower temperature, and before the remaining syrup has been diluted by the separation of the anhydrous crystals, concentrated solutions are rather viscous and this viscosity prevents a free crystallization. A good temperature is 30 to 40° C. The time within which the crystallization is completed varies between half a day and several weeks, according to the purity of the mass.

Though it is always well, in order to secure a uniform and speedy crystallization, to start it by the introduction of some crystals, yet it is possible and, for sugars of high purity, quite easy to obtain the same crystallization by simply keeping the concentrated solutions at a temperature of about 30° C for some time. Under these circumstances a crystallization of anhydrous grape sugar takes place. This behavior of grape sugar is also unexpected. Soxhlet, who, a short time ago, took patents in different countries for the refining of grape sugar by means of alcoholic liquids, and for the production of a hard crystallized grape sugar, describes one of his products expressly as the hydrate of the formula  $C_6H_{12}O_6 \cdot H_2O$ , yet he concentrates highly a solution of very pure grape sugar and allows it to crystallize at an elevated temperature. I have failed, under the conditions of my experiments, to obtain the hydrate. But that it is possible for the hydrate to crystallize in large and well developed crystals has been established in 1877 by Halse and Steiner, who analyzed a crystallized hydrate of grape sugar, of which some crystals weighed 4 to 5 grams, and which was readily taken for cane sugar. This grape sugar had made the voyage from England to Australia

and back and during this time had undergone the transformation.

A product which has for some time played an important part in the literature of this subject is Anthon's hard crystallized grape-sugar. As early as 1857, Anthon in Prague prepared a very pure sugar by crystallizing and pressing the hydrate; he then melted the press-cakes without addition of water, and allowed the mass to solidify in moulds.

He obtained crystalline masses which, according to his analysis, contained 4.7% of water and for which he claimed the constitution of a half hydrate of grape sugar of the formula,  $2(C_6H_{12}O_6) + H_2O$ . As he did not drain his crystals, he certainly had nothing but a mixture of anhydrous sugar and the hydrate, the surplus water of the hydrate having been evaporated during the melting. This has already been suggested by Stohmann in the latest German edition of Muspratt's chemistry (vi 2077).

Crystallized anhydrous grape sugar such as I have prepared it from a watery solution has the following properties. Dried at 30 to 40° C. it does not retain more than .02 per cent. of moisture; the moisture determination made at 130° C. It shows a neutral reaction, with sensitive litmus paper. It melts in a capillary tube between 141 and 145° C. It was tested in the polariscope, and showed birotation. Landolt in his book on the optical rotatory power of organic substances (Braunschweig 1879, p. 184), gives 32.68 grams as the amount of pure grape sugar, which taken instead of the normal weight of cane sugar should show 100 on the scale of a Ventzke-Soleil instrument.

Mr. Lungwitz, who has made these determinations for me, found, if he rapidly dissolved this amount in cold water and immediately polarized the solution, a polarization varying between 202 and 204; if he allowed it to stand for 24 hours, 101 to 102.

This difference is mainly due to an error in Landolt's figure. This figure is calculated from an assumed specific rotation of  $a_D = 53.0$ . This is correct only for a concentration of 10 grams of sugar in 100 cc. of solution, but for a concentration of 32.68 grams in 100 cc.  $a_D$  becomes  $= 53.57$  according to Tollen's determinations. Therefore 32.68 grams ought to polarize 101.1; while the observed polarization for monorotation was 101 to 102.

These are the facts so far as they refer to chemistry, but in view of the increasing importance of grape sugar as an article of general consumption, I wish to add a few remarks with reference to the industrial application of these observations.

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In the ordinary process of the manufacture of grape sugar from starch the conditions are such that the resulting product is always far from being pure grape sugar, however pure the starch from which it is derived may have been. Though a good method for the quantitative determination of starch consists in its conversion with a mineral acid and subsequent determination with Fehling's solution, yet, in practice, a smooth and complete conversion is not attainable. The reason of this difference lies in the fact that the chemist, for a complete conversion, works with a very diluted solution, while the manufacturer necessarily works with solutions of higher density. At a higher density, however, the acid seems to act on the sugar already formed, and before all the dextrine is converted into sugar, the sugar itself is partially converted into something else which constitutes an impurity in the final product. So far we know very little about the nature of these impurities of commercial grape sugar, but several chemists have asserted that the residues which remain after fermentation and distillation are more or less injurious to the human system. This subject, though, requires a more complete investigation. As the principal use of all the grape sugar produced is that which is made of it in the manufacture of fermented beverages, beer and wine, it is easy to understand the rising demand for a purer article.

Fr. Anthon has, twenty years ago, called attention to the disadvantages arising from the use of impure grape sugar in wine making, and suggested a remedy. His suggestion was to refine the ordinary grape sugar by crystallization and the use of a centrifugal machine for the removal of the liquid impurities. He modified this process in so far as he used a strong press instead of a centrifugal machine, and, according to the testimony of several chemists, really produced an article of remarkable purity. His process seems to have never been used for any length of time or on an extensive scale.

Fouchard had already, in 1853, manufactured a refined grape sugar by allowing grape sugar solutions to crystallize in barrels and then withdrawing the liquid portion through a number of holes in the bottoms of the barrels.

Though the principle of these refining processes is correct, yet there is a difficulty inherent in it which arises from the form and nature of the crystals in which the sugar solidifies. Under ordinary circumstances grape sugar crystallizes from a watery solution as the hydrate in the shape of very fine tablets which are mostly

## THE WATER SUPPLY OF THE CITY OF NEW YORK.

grouped spherically. Owing to the fineness of the tablets and the capillary attraction, it is difficult to remove the impure mother-liquor sufficiently from the crystals by means of a centrifugal machine, and even with a hydraulic press high purity cannot be obtained, together with a large yield. It is different with the crystals of anhydrous grape sugar. They are of a prismatic shape, and form loose aggregations from which the syrup can be easily removed by centrifugal force, and which lend themselves to a treatment of draining and washing very similar to that of cane sugar. Under these circumstances it is possible to produce a grape sugar which compares in purity with block and granulated cane sugar. A number of applications for such an article readily suggest themselves. The confectioner, the druggist, the manufacturer of condensed milk may use it. In the preparation of certain wines it can safely take the place of cane sugar; but its principal use ought to be in the kitchen for all those preparations where utmost sweetness is not sought for. It is not so well suited for sweetening tea or coffee, though it does not quite so unfavorably compare with cane sugar as the books will have it. To obtain a moderate sweetness, equal to that produced by a given amount of cane sugar, it is not necessary to take  $2\frac{1}{2}$  or 3 times as much as cane sugar, but only about  $1\frac{1}{3}$  times the quantity; at least I have found it so, and some of my friends also.

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## V. THE WATER SUPPLY OF THE CITY OF NEW YORK.

BY E. WALLER, PH. D.

I desire, in the first place, to present the results of complete analyses of the Croton water made at different times. The various denominations of salts quoted have been given in order to literally quote the different analysts. For the three first, double columns are given, representing the results in grains per English (Imperial) gallon of 70,000 grains, and also in grains per United States gallon of 58,318 grains, the first columns in each case being the form in which the analysts have recorded their results, to judge from the context. In Nos. 4 and 5, the magnesium and calcium bicarbonate have been calculated back to mono-carbonates, and the results given in brackets. Another table of the same results, calculated to parts per 100,000, is appended.



There is probably less difference in the results than the figures would seem to indicate, the mode of stating the results in the earlier analyses rather suggesting different methods of conducting the examination, and calculating the results to those at present in use.

Next, permit me to call your attention to a chart showing graphically the variations found in the constitution of the Croton water by Dr. C. F. Chandler during the Summer months of 1867 and 1868, representing some fifty examinations, and my own results, made in a similar manner every week, from the latter part of 1872 to the middle of 1879, representing about 350 examinations. The average results may be thus stated :

Average of results of tests made on Croton water (Parts per 100,000) :

		Mineral matter.	Org. and volatile.	Total solids.	Hardness.	Ox'n absor- bed from Permanganate.
Summer of	1867	6.72	1.12	7.84	4.32	0.181
"	1868	5.66	1.97	7.63		0.168
Last 2 mos.	1872	7.48	0.44	7.92	3.553	0.131
Year	1873	6.23	1.59	7.82	3.395	0.135
"	1874	5.83	1.76	7.59	3.332	0.166
"	1875	5.656	1.835	7.491	3.293	0.211
"	1876	5.416	1.682	7.098	3.159	0.185
"	1877	5.603	1.823	7.426	3.260	0.253
"	1878	5.299	1.904	7.203	2.846	0.183
First 5 mos.	1879	5.424	0.912	6.336	2.811	0.072
Average from Nov. 1872, to May, 1879, inclusive		5.702	1.678	7.380	3.210	0.180

The "Total solids" were determined by weighing the residue obtained by evaporating a measured quantity ; the "Organic and Volatile" by igniting this residue, moistening with carbonic acid water, drying and weighing again ; "Hardness" by standard soap solution as usual, the results being expressed in the equivalent of calcium carbonate, while the "Permanganate" test was that of Miller. (J., Lond. Chem. Soc., 1865, p. 117 ; see also R. Angus Smith, Watts' Dictionary, v. 1029 ; Wauklyn's Water Anal., 1st Ed., 1868, p. 42. &c. and others), acidifying with  $H_2SO_4$ , adding



standardized solution of permanganate, until the color held for half an hour at the ordinary temperature of the laboratory.

In addition to these, several other examinations made at irregular intervals, and at other times than those specified above might be given, but as they present no marked deviations, I will not occupy your time with them.

As a sample of similar determinations made on samples of the Croton, taken from different parts of the city at the same time, I would present the results obtained in April of last year, when the odors in the water caused suspicion that it contained some compounds dangerous to health.

No.	Locality.	Mineral matter.	Organic & Vol.	Total Solids.	Oxygen absorbed from Permanganate.
1	West 33d St.	6.6	2.2	8.8	0.064
2	East 34th St.	6.0	1.5	7.5	0.060 F
5	West 131st St.	4.4	1.3	5.7	0.064 F
8	East 122d St.	5.7	trace	5.7	0.062

The samples marked F were clarified by subsidence or filtration before examination, as they contained varying amounts of muddy sediment, and were therefore not fair samples of the water as ordinarily used. It may be mentioned that about a pint of No. 5 on standing half an hour in a cylinder, some 2½ inches in diameter, deposited a sediment ⅔ of an inch in depth. When this sediment was distributed as evenly as possible through the water and a portion was examined, the results were

No.	Mineral.	Org. and Vol.	Total.	Oxygen from Permanganate.
2	75.7	21	96.70	0.366
5	69.2	15.1	84.30	0.455

A portion of the sediment was examined separately. Shaken with ether it afforded as soluble in that menstruum, a minute proportion of vegetable wax having a slight greenish-brown tinge, probably from the presence of chlorophyll.

A small amount was obtained for analysis. The results were

Loss on ignition.	23.31		
Silica	43.61 to 51.00%		
Lime	0.63	=CaCo <sub>3</sub>	1.12%
Magnesia	3.16	=MgCo <sub>3</sub>	6.64

Ferric and Aluminic oxides 20.92

As to other determinations on the Croton Water, the following results are offered.

Parts per 100,000.

Date.	Free Ammonia.	Albummoid Ammonia.	Remarks.
August, 1874.	0.00095	0.0145	average of 6
December, 1877.	0.0010	0.0102	" of 2
Nov. 16th, 1878.	0.0015	0.0130	
July 11th, 1879.	0.0008	0.008	
April 4th, 1881.	0.0020	0.0110	
" 22d, "	0.0016	0.0117	average of 10
" " "		0.019 to 0.031	Total NH <sub>3</sub> on very turbid samples.
May 16th, 1881.	0.001	0.007	
Nov. 8th, 1881.	0.002	0 012	
Nitrogen in Nitrates.			
July 29th, 1881.	0.0198		
Nov. 8th, "	0.0181		

The results obtained on free and albuminoid ammonia do not indicate any material alteration in the proportions of those constituents yielding nitrogen in those forms since the examinations began to be made. The investigation of last April shows that the quality of the water does not vary very much in different parts of the city at the same time, unless the sediment is mixed in, when the amount of nitrogen obtainable as ammonia by distillation may reach nearly thrice the amount obtainable from the sample when fairly clear.

The amounts of nitrogen in nitrates so far as they go give no indications of sewage contamination in the water.

About the end of last year a paper by Prof. Leeds on "Relative purity of city waters in the United States," was published in the *Chemical News* (XLIV p. 265), in which the Croton water was condemned as contaminated.

The analytical results were given as follows:—

Croton, June 23d, 1881.		(Results in parts per 100,000)	
Free Ammonia	0.0027	Chlorine	0.350
Albuminoid "	0.027	Hardness	3.30
Oxygen required	0.81	Total Solids	11.80
Nitrites	none	Mineral Matter	5.00
Nitrates	0.8325	Organic & Vol.	6.80

These results I strenuously object to as misleading.

The term "nitrates" is indefinite, and when so many chemists calculate their results to "nitrogen in nitrates," &c. a false impression is readily created by the above statement in that regard. The "oxygen required," I have learned was obtained by Kubel's method—by the action of potassium permanganate on the water strongly acidified with sulphuric acid at the boiling temperature. Inasmuch as most chemists (at least in English speaking countries), use the permanganate test at ordinary temperatures, such a statement as the above, without specifying the method used, is calculated to convey a false impression of the quality of the water. Moreover the test performed in that way is open to serious objections.

Under those circumstances the chlorine in the water would affect the results, and Prof. Leeds himself has shown us that the reagents used invariably contain impurities which would affect the test to the prejudice of the water tested, the permanganate containing chlorine compounds, and the sulphuric acid (which is used in considerable amount) containing lower oxides of nitrogen and sulphur, so that where a line is drawn on comparatively small amounts of oxygen absorbed from permanganate, the impurities in the reagents would make a great difference in the conclusions to be drawn.

Prof. Leeds' results on Total Solids and Free and Albuminoid ammonia are very high, indeed higher than any results I have obtained during the past fourteen years, except when—as last spring—the samples of water were so charged with sediment as to render them by no means fair samples of the Croton Water as ordinarily obtainable. The conclusion would seem to be that his sample was turbid with sediment.

With regard to other determinations they either agree with the preceding examinations, or the methods employed were different from those of which I made use, and therefore preclude a comparison between them.

In commenting on the results, Prof. Leeds remarks: "New York and all the places mentioned lower on the list receive their water from contaminated sources. The feeders which empty into Croton Lake, the principal reservoir of the New York water, pass through a settled country, with numerous tanneries, factories, &c., along their banks. Analyses of the Croton water, made at different times during the past five years, have shown that it is to be classed among contaminated water supplies." A quotation of this statement was sent to Mr. Isaac Newton, Chief Engineer of the Croton Aqueduct Department. His reply was briefly to the effect that he

had comparatively recently examined the Croton water-shed, and that Prof. Leeds' assertion with regard to it was altogether erroneous.

From other sources, I have been able to ascertain, *first*, that with regard to the population of the water-shed: for its area of 339 square miles the population is from 17 to 20,000, or about one man to every ten acres. Permit me to quote the table given by Mr. D. M. Greene in the 23d annual report of Water Commissioners of Troy for 1877, p. 120.

Population of water sheds for city supplies :

City.	Population per square mile.
Rochester, N. Y. . . . .	36
New York, N. Y. . . . .	65
Albany, N. Y. . . . .	77
Poughkeepsie, N. Y. . . . .	86
Schnectady, Cohoes, and West Troy, N. Y. { Supply from Mohawk River. . . . .	103
Brooklyn, N. Y. . . . .	119
Boston, Mass., . . . . .	229
London, England, . . . . .	270

*Second.*—As to industries in the Croton water-shed. But few tanneries now exist in that region for the simple reason that the most of the trees yielding the necessary bark have been cut down, and tanning is no longer profitable in that section. As regards other industries the region contains but few factories of any kind, and those are on a small scale.

To sum up, I desire to express a most emphatic dissent from Prof. Leeds' conclusions for the following reasons:

1. The proportion of chlorides existing in the water has not increased of late years so far as the records extend, and hence no indications of contamination by sewage or manufacturers can be asserted to exist.

2. The amounts of oxygen absorbed by permanganate test for a number of years, (serving to compare the water with itself at different times) show no changes in the quality of the Croton water. The same may be said for the results on Free and Alluminoid Ammonia and Organic and Volatile matter.

3. The Croton water-shed is not crowded either with population or with manufacturers as Prof Leeds seems to imagine.

4. The health of the community is not, and has never been, such as to indicate the presence of any contamination in the water supply.

COMPLETE ANALYSES OF CROTON WATER.  
RESULTS IN GRAINS PER GALLON.

Number.....	1 1		2 2		3 3		4	5	6	7
Analyst .....	Professor J. C. Booth.		Dr. J. R. Chilton.				C.F.Chandler.		E. Waller.	
Date.....	1843.		1843.		August, 1859.		Sum'er 1869.	May, 1872.	May, 1879.	Nov. 1881.
Gallon used.....	Eng.	U. S.	Eng.	U. S.	Eng.	U. S.*				
Sodium chloride.....	.....	.....	0.44	0.367	4.404	0.336	0.402	0.284	0.205	0.205
Calcium sulphate.....	.....	.....	.....	.....	0.853	0.294	0.158	0.021	0.723	.....
Alkaline chlorides.....	0.198	0.161	.....	.....	.....	.....	.....	.....	.....	.....
Potassium sulphate.....	.....	.....	.....	.....	.....	.....	0.179	0.205	0.188	0.201
Sodium sulphate.....	.....	.....	.....	.....	.....	.....	0.260	0.024	0.200	0.216
Akaline carbonates.....	0.828	0.690	0.90	.....	0.270	0.225	.....	.....	.....	0.054
Magnesium chloride....	.....	.....	0.84	0.750	0.147	0.122	.....	.....	.....	.....
Calcium chloride.....	.....	.....	.....	.....	0.104	0.087	.....	.....	.....	.....
Magnesium carbonate..	0.930	0.782	1.52	0.700	0.390	0.325	(1.101)	(0.770)	0.918	0.685
Calcium carbonate.. ...	2.298	1.910	.....	1.266	0.836	0.696	(1.648)	(1.439)	1.650	1.319
Magnesium bicarbonate	.....	.....	.....	.....	.....	.....	1.913	1.338	.....	.....
Calcium bicarbonate....	.....	.....	.....	.....	.....	.....	2.670	2.331	.....	.....
Ferric & aluminic oxides	0.110	0.092	.....	.....	.....	.....	trace	0.058	0.175	0.045
Silica.....	0.359	0.299	0.46	0.383	0.170	0.142	0.021	0.222	0.274	0.210
Organic and volatile....	0.276	0.240	.....	.....	0.916	0.763	0.670	0.874	0.560	0.233
Total solids. ....	.....	.....	.....	.....	3.590	2.990	6.873	5.360	.....	.....
Solids by evaporation...	4.998	4.174	4.16	3.466	3.705	3.087	4.780	3.849	4.893	3.168
Chlorine.....	.....	.....	.....	.....	.....	0.296	0.243	0.172	0.121	0.124

ABOVE RESULTS GIVEN IN PARTS PER HUNDRED THOUSAND.

Number.....	1	2	3	4	5	6	7
Analyst.....	Booth.	Chilton.		Chandler.		Waller.	
Date.....	1843.	1843.	1859.	1869.	1872.	1879.	1881.
Sodium Chloride.....	0.276	0.629	0.577	0.690	0.487	0.351	0.351
Calcium sulphate.....			0.504	0.272	0.041	1.239	
Alkaline chlorides.....							
Potassium sulphate.....				0.309	0.351	0.322	0.345
Sodium sulphate.....				0.449	0.041	0.343	0.371
Alkaline carbonates.....	1.183		0.386				0.092
Magnesium chloride.....		1.286	0.210				
Calcium chloride.....			0.149				
Magnesium carbonate....	1.341	1.200	0.557	(1.898)	(1.320)	1.575	1.174
Calcium carbonate.....	3.276	2.171	0.194	(2.826)	(2.467)	2.830	2.262
Magnesium bicarbonate.....				3.280	2.294		
Calcium bicarbonate.....				4.578	3.996		
Ferric and aluminic oxides.....	0.157	0.657	0.243	trace.	0.100	0.300	0.078
Silica.....	0.513						
Organic and volatile..	0.394			1.060	0.380	0.470	3.360
			1.309	1.150	1.500	0.940	0.400
Total.....			5.129	11.788	9.190		
Solids by evaporation.....	7.140	5.943	5.293	8.200	6.000	8.890	5.432
Chlorine.....			0.508	0.416	0.294	0.213	0.213

Nos. 1 and 2. Illustrations of the Croton Aqueduct. F. B. Tower. N. Y., 1843, p. 135.  
No. 3. Report of Water Commissioners of Albany for 1865, p. 50.  
Nos. 4 and 5. Report of Board of Health for 1871, New York, p. 371.  
No. 6. Report on Croton Water, New York, 1881, p. 43.  
\* U. S. gallon of 231 cubic inches (56,318 grains).

## VI. HEPTYLENE FROM HEPTANE OF P. SABINIANA.

By F. P. VENABLE, PH. D.

University of North Carolina.

In a note on certain heptylic derivatives published in 1880\*, mention was made of a heptylene agreeing in boiling point with the one prepared by Schorlemmer from the petroleum-heptane. This heptylene was observed as one of the products in preparing several of the heptylic derivatives from the bromide, but the method adopted for its special preparation was the action of sodium ethylate upon the heptylic bromide at the temperature of boiling. The liquid obtained was light, mobile and with a high refractive-index, boiling at 97°–98°C, and possessing a pleasant aromatic smell. Its specific gravity was 0.70075 (21°. 6 C).

If sodium ethylate is allowed to act in the cold, a heptylethyl-ether is formed, together with a small amount of heptylene. Even when an excess of the sodium ethylate is present, it is difficult to change all of the bromide to the ether. Analyses, however, of the heavy oily liquid obtained showed the conversion to be almost complete

## HEPTYLENE BROMIDE.

The action of bromine upon the heptane is slow, and heat must be applied. On the heptylene, bromine acts with great energy, heat being evolved. In the first case, the resulting product is one of substitution; in the latter, it is one of addition; but substitution products are very easily formed if the temperature is allowed to rise. In the brominating, then, it is necessary to avoid all rise of temperature. The heptylene was put in a long tube surrounded by a freezing mixture of sodium sulphate and hydrochloric acid, and air loaded with bromine vapor was sucked through it for six or ten hours. The color of the bromine disappeared with each bubble, and the reaction was stopped as soon as a permanent coloration was observed. The thick, oily liquid was then distilled in partial vacuum, boiling quite constantly without the evolution of hydrobromic acid gas. After the first distillation it was found possible to complete the fractioning under ordinary pressure. The boiling point was 209°–211° C. (uncorrected.)

Only with great difficulty did Schorlemmer † succeed in

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\* Ber. Chem. Ges. XIII. 1649.

† Annalen Chem. Pharm. 188, 286.

forming a heptylene bromide or heptylic dibromide from his petroleum-heptane, and it could not be distilled. The dibromide prepared by Thorpe and Young ‡ has a spec. grav. 1.5146 (18.5° C), and underwent decomposition at the temperature of 150° C—that is, considerably below the boiling point as observed for the dibromide described above.

I propose carrying the investigation of this heptylene and its derivatives still further, as their stability gives promise of interesting results. A careful determination of the specific gravity of the the dibromide will also be made as soon as a larger supply is on hand.

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‡ *Annalen Chem. Pharm.* 165, 12.

# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Room 1, University Building, N. Y., March 3d, 1882.*

The meeting was called to order at 8:20 p. m. Prof. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

No report was received from the Board of Directors.

Dr. E. Hirshberg was then elected a regular member.

After which the following gentlemen were nominated as members of the Society:

Thos. S. Gladding as regular member. Proposed by Chas. M. Stillwell, T. J. Parker and A. R. Leeds.

Dr. G. Krause as regular member. Proposed by Geo. A. Prochazka, A. R. Leeds, and James H. Stebbins, Jr.

Robert G. Remsen, Jr., M. D., as regular member. Proposed by A. R. Leeds, James H. Stebbins, Jr., and A. H. Elliott.

Prof. A. A. Brennerman as regular member. Proposed by A. R. Leeds, James H. Stebbins, Jr., and A. H. Elliott.

G. E. Perkins as regular member. Proposed by T. O'C. Sloane, M. Benjamin and W. Rupp.

After which Mr. Elliott moved that the regular order of business be suspended, in order to give more time to discuss the publishing of the Journal.

Carried.

The Secretary then moved that the above discussion be postponed to 9 p. m., in order that some of the papers of the evening might be read.

Carried.

The first paper of the evening: "On the Determination of Zinc as Pyrophosphate," by Mr. Geo. C. Stone, was then read.

After some remarks by the Chairman, Dr. Sloane read his first paper "On Methyl Violet Test Paper."

Some remarks by Dr. Grothe followed.

After which Dr. Sloane read his second paper: "On a Fat Extraction Apparatus."

The third paper: "Upon the Separation of Alumina and Sesquioxide of Iron from Manganese," by Mr. Nelson H. Darton, then followed.

Dr. Endemann stated that Drs. Behr and Ledoux had both



resigned from the Committee on Papers and Publications, and that he now, officially, tendered his own resignation as Editor; but stated that he would remain Editor till the next number of the Journal is published.

He furthermore stated that he did not think it would be wise to increase the Committee on Papers and Publications to five, as he thought three competent men would be sufficient.

After a good many remarks, both for and against the proposed change, it was finally moved that this subject be referred to the Board of Directors, who in turn shall recommend their decision to the Society.

Carried.

After which it was moved that the reading of the papers should be proceeded with.

Carried.

Dr. Waller then took the chair.

The fourth paper: "On Some New Compounds of Diphenylamine," by Prof. A. R. Leeds, was then read.

After which the fifth paper: "On Methods of Water Analysis and the Character of the New York Water Supply," by Prof. A. R. Leeds was read.

Dr. Waller remarked that he also considered the permanganate method of Mille as unsatisfactory when applied to isolated samples, but that he considered it of value when the same water is compared with itself at different times. He also objected to Kubel's method, on the grounds previously mentioned in his paper. He did not mean to criticize unfavorably Dr. Leeds method for "mineral matter," but preferred to use carbonic acid water after ignition.

As the hour was late it was moved that the paper of Mr. Gladding on Reverted Phosphates be made a special order of business at the next Conversazione.

Carried.

After which the meeting adjourned.

JAMES H. STEBBINS, JR.,  
*Recording Secretary.*

## VII. DETERMINATION OF ZINC AS PYROPHOSPHATE.

BY GEO. C. STONE, PH. B.

Sometime since I had occasion to analyze a sample of water said to contain the tailings from an amalgamating mill. The analysis of the suspended matter only added up about eighty-eight per cent. I tried boiling the filtrate from the magnesia with KOH, this threw down a fine, crystalline, white precipitate; on making a qualitative examination of this I found it contained ZnO, and  $P_2O_5$ . On weighing the precipitate and calculating the amount of ZnO it contained, supposing it to be  $Zn_2 P_2 O_7$ , I found it eleven and a fraction per cent., which made my analysis add up very close to one hundred per cent. This led me to experiment with phosphate of zinc with the following results.

If a mixture of an acid solution of a zinc salt and an alkaline ortho-phosphate is exactly neutralized with ammonia, the zinc is precipitated as a bulky, flocculent double phosphate of zinc and ammonia, easily soluble in acids or ammonia, and slightly soluble in potassic hydrate. If the solution in ammonia is boiled, the double phosphate reprecipitates as a fine, crystalline white precipitate, very slightly soluble in ammonia, and almost absolutely insoluble in water. If the boiling is continued till the free ammonia is all driven off, the precipitation is complete. On drying and igniting this precipitate the zinc is obtained as pyrophosphate  $Zn_2 P_2 O_7$ .

This method of determining zinc has several advantages over the one commonly employed, of precipitating as sulphide, dissolving in hydrochloric acid, reprecipitating as basic carbonate, igniting and weighing as oxide.

First.—It is much quicker, as it is not necessary to let the precipitation stand, and there is only one precipitation.

Second.—The precipitate settles rapidly, filters quickly, does not run through the filter, washes easily, dries quickly, brushes off the paper easily and completely when dry, and is very difficult to reduce.

Third.—The presence of ammonia salts does not interfere with the completeness of the precipitation.

In all of which particulars its behavior is quite the reverse of that of either the sulphide or basic carbonate.

Fourth.—The pyrophosphate contains only 42.76 per cent. of zinc, while the oxide contains 80.25 per cent. so that an error in weighing makes much less difference in the result.

Fifth.—The phosphate does not need any purification, whereas the oxide frequently contains silica.

On the other hand the phosphate bumps badly while boiling out the ammonia; and the precipitate sticks pretty tightly to the beaker. If the solution is kept rather dilute and only just enough ammonia is added to redissolve the precipitate first formed, and the solution is frequently stirred, the bumping will not give much trouble. The precipitate that sticks to the beaker is best removed by dissolving in a few drops of HCl, adding a little  $\text{NaNH}_4\text{HPO}_4$ , making just alkaline with  $\text{NH}_4\text{OH}$  and boiling till the free ammonia is expelled.

*Experiment I.*—Was made more to test the behavior of the phosphate of zinc and to see if the results agreed, than to test the absolute accuracy of the method. 0.2 grms. of zinc, coated with oxide, were dissolved in  $\text{H}_2\text{SO}_4$  and a few drops of  $\text{HNO}_3$ , the solution was diluted to about 300 cc., an excess of  $\text{NaNH}_4\text{HPO}_4$  added, and just enough  $\text{NH}_4\text{OH}$  to re-dissolve the precipitate first formed. The solution was then boiled till there was no longer any smell of  $\text{NH}_3$ , filtered, washed thoroughly with hot water, dried, ignited and weighed. Gave :

A  $\text{Zn}_2\text{P}_2\text{O}_7$  0.4627 grms. = Zn 0.1979 grms.

B  $\text{Zn}_2\text{P}_2\text{O}_7$  0.4624 grms. = Zn 0.1977 grms.

A was filtered as soon as the  $\text{NH}_3$  was expelled. B was allowed to stand in a warm place for three hours after boiling.

*Experiment II.*—0.2 grms. of zinc ore, mainly oxide, was dissolved in HCl,  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$ , evaporated till the HCl and  $\text{HNO}_3$  were expelled, diluted, filtered, the iron separated as basic acetate, dissolved and reprecipitated by a large excess of  $\text{NH}_4\text{OH}$ , the filtrates combined and evaporated to about 300 cc., an excess of  $\text{NaNH}_4\text{HPO}_4$  added and the zinc determined as in I. In a second portion of 1.0 gm. the iron was separated as above, the zinc precipitated by  $\text{H}_2\text{S}$  in a boiling acetic acid solution, let stand, filtered, washed, dissolved in HCl, reprecipitated as basic carbonate by  $\text{Na}_2\text{CO}_3$ , ignited and weighed as oxide. Gave :

A  $\text{Zn}_2\text{P}_2\text{O}_7$  0.3441 grms. Zn 0.1471 grms. = 73.55 per cent

B  $\text{Zn}_2\text{P}_2\text{O}_7$  0.9149 grms. Zn 0.7341 grms. = 73.41 “ “

*Experiment III.*—0.2 Grms. ferruginous blende treated as in II and gave :

A  $\text{Zn}_2\text{P}_2\text{O}_7$  0.2721 grms. = Zn 58.17 per cent

B  $\text{Zn}_2\text{P}_2\text{O}_7$  0.2716 grms. = Zn 58.07 “ “

The analysis was afterwards completed and gave :

Zn (average of 2 determinations)	58.12 per cent
Fe " " "	7.61 " "
Insoluble silicious matter	1.43 " "
S calculated to give Fe S and Zn S	32.96 " "
<hr/>	
Total,	100.12

*Experiment IV.*—1.0 gram.  $\text{Zn} (\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2 \text{H}_2\text{O}$  dissolved in water, excess of  $\text{NaNH}_4\text{HPO}_4$  and  $\text{NH}_4\text{OH}$  added, the  $\text{NH}_3$  expelled by boiling and the zinc determined as before. Gave :

A	$\text{Zn}_2\text{P}_2\text{O}_7$	0.6953 grms.	= Zn 29.78 per cent
B	$\text{Zn}_2\text{P}_2\text{O}_7$	0.6974 grms.	= Zn 29.82 " "

The theoretical percentage is 29.68.

*Experiment V.*—To test the influence of alkaline and ammonia salts :

0.5 grms.  $\text{Zn} (\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2 \text{H}_2\text{O}$ , 2.0 grms.  $\text{Na}_2\text{CO}_3$ , 2.0 grms.  $\text{NH}_4\text{Cl}$ , and 5.0 grms.  $\text{Na} \text{C}_2\text{H}_3\text{O}_2$  the zinc determined as before gave :

A	$\text{Zn}_2\text{P}_2\text{O}_7$	0.3484 grms.	= Zn 29.78 per cent.
B	$\text{Zn}_2\text{P}_2\text{O}_7$	0.3469 grms.	= Zn 29.66 per cent.

*Experiment VI.*—As the phosphate of zinc is soluble in  $\text{NH}_4\text{OH}$ , while the phosphates of lime, magnesia, and manganese are insoluble in it, it seemed probable that the zinc could be separated by means of this reaction. The following experiments were tried to test this: 0.5 gram.  $\text{Zn} (\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2 \text{H}_2\text{O}$ , 0.1 gram.  $\text{Mn}_2\text{O}_3$ , 0.1 gram.  $\text{CaCO}_3$  and 0.1 gram.  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  were dissolved in  $\text{HCl}$  excess of  $\text{Na} \text{NH}_4\text{HPO}_4$  and  $\text{NH}_4\text{OH}$  added and let stand cold for eighteen hours, the clear solution filtered off, the precipitate dissolved in  $\text{HCl}$ , reprecipitated by  $\text{NH}_4\text{OH}$  and a few drops of  $\text{Na} \text{NH}_4\text{HPO}_4$ , filtered, washed with cold dilute  $\text{NH}_4\text{OH}$ , the  $\text{NH}_3$  boiled out, and the zinc determined as before, gave:

A	$\text{Zn}_2\text{P}_2\text{O}_7$	0.3444 grms.	= Zn 29.46 per cent.
B	$\text{Zn}_2\text{P}_2\text{O}_7$	0.3451 grms.	= Zn 29.52 per cent.

The phosphates of manganese and lime clogged the filter and showed a tendency to run through it. To get good results the solution must be rather dilute (400 to 500 cc.).

*Experiment VII.*—0.5 grms.  $\text{Zn} (\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$  and 0.5 grms.  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ , treated as in the last experiment, gave:

A	$\text{Zn}_2\text{P}_2\text{O}_7$	0.3453	= Zn 29.54 per cent.
B	$\text{Zn}_2\text{P}_2\text{O}_7$	0.3451	= Zn 29.52 per cent.

*Experiment VIII.*—As phosphate of lime filters very slowly and is very difficult to wash, I tried precipitating the lime as oxalate in a rather dilute solution, decanting, dissolving in HCl, reprecipitating, and determining the zinc in the filtrate as phosphate. 0.5 grms.  $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$  and 0.25 grms.  $\text{CaCO}_3$  taken, gave:

$$\text{Zn}_2\text{P}_2\text{O}_7 \text{ 0.3429 grms.} = 29.32 \text{ per cent.}$$

*Experiment IX.*—1.365 grms.  $\text{Zn}_2\text{P}_2\text{O}_7$ , heated to redness for about half an hour, weighed 1.365 grms.

*Experiment X.*—0.4 grms. zinc ore, containing about 14 per cent.  $\text{SiO}_2$ , 20 per cent.  $\text{Fe}_2\text{O}_3$ , 12 per cent.  $\text{CaCO}_3$ , and 16 per cent.  $\text{MnO}$ . A. Iron separated as basic acetate, dissolved and reprecipitated. Manganese precipitated by Br. Excess of  $\text{NaNH}_4\text{HPO}_4$  and  $\text{NH}_4\text{OH}$  added, the solution decanted from the lime, precipitate, dissolved and reprecipitated by a large excess of  $\text{NH}_4\text{OH}$ , filtered and washed with dilute  $\text{NH}_4\text{OH}$ , using a filter pump, the excess of  $\text{NH}_3$  boiled out, and the zinc ignited and weighed as  $\text{Zn}_2\text{P}_2\text{O}_7$ .

$$\text{A. } \text{Zn}_2\text{P}_2\text{O}_7 \text{ 0.2760, Zn 0.1179} = 29.47 \text{ per cent.}$$

B. Iron precipitated as basic acetate, dissolved and reprecipitated by a large excess of  $\text{NH}_4\text{OH}$ , the filtrates combined, made strongly acid with  $\text{HC}_2\text{H}_3\text{O}_2$ , the zinc precipitated by  $\text{H}_2\text{S}$  in a boiling solution, filtered, dissolved in HCl and  $\text{KClO}_3$ , reprecipitated by  $\text{Na}_2\text{CO}_3$ , ignited, and weighed as  $\text{ZnO}$ , gave:

$$\text{B. } \text{ZnO} \text{ 0.1466, Zn 0.1176} = 29.40 \text{ per cent.}$$

*Experiment XI.*—0.4 grms. ore similar to the last, treated in the same way, gave:

$$\text{A. } \text{Zn}_2\text{P}_2\text{O}_7 \text{ 0.2468 Zn 0.1055} = 26.37 \text{ per cent.}$$

$$\text{B. } \text{ZnO} \text{ 0.1303 Zn 0.1045} = 26.15 \text{ per cent.}$$

*Experiment XII.*—0.4 grms. ore similar to the last, treated in the same manner, gave:

$$\text{A. } \text{Zn}_2\text{P}_2\text{O}_7 \text{ 0.2390, Zn 0.1022} = 25.55 \text{ per cent.}$$

$$\text{B. } \text{ZnO} \text{ 0.1294, Zn 0.1030} = 25.75 \text{ per cent.}$$

The results of the separations are not as good as I could wish; but I intend to continue the experiments, in hopes of improving on them.

## RESUME:

	Substance Ana- lyzed.	Grms. used.	Grms Zn found.	REMARKS.
Expt. I.	Impure Zinc	0.2	0.1979	
	" "	0.2	0.1977	
" II.	Zinc Ore	0.2	0.1471	Weighed as $Zn_2P_2O_7$
	" "	1.0	0.7341	" $ZnO$
" III.	Blende	0.2	0.11634	
	" "	0.2	0.11614	
" IV.	$Zn(C_2H_3O_2)_2$	0.1	0.2978	Theory requires 0.2968
	$2H_2O$	0.1	0.2982	grms Zn
" V.	"	0.5	0.1489	+2 grms. $NH_4Cl$ + 2 grms. $Na_2CO_3$ + 5 grms. $Na_2C_2H_3O_2$
" VI.	"	0.5	0.1484	+0.1 gm. $Mn_2O_3$ + 0.1 gm. $CaCO_3$ + 0.1 gm. $MgSO_4 \cdot 7H_2O$
" VII.	"	0.5	0.1476	+0.5 grms. $MgSO_4 \cdot 7H_2O$
" VIII.	"	0.5	0.1477	+0.35 grms. $CaCO_3$ Separated as oxalate.
" IX.	$Zn_2P_2O_7$	1.365	1.865	Ignited for half an hour.
	Zinc Ore.	0.4	0.1179	Weighed as $Zn_2P_2O_7$
" X.	" "	0.4	0.1176	" $ZnO$
" XI.	" "	0.4	0.1055	" $Zn_2P_2O_7$
	" "	0.4	0.1046	" $ZnO$
" XII.	" "	0.4	0.1033	" $Zn_2P_2O_7$
	" "	0.4	0.1030	" $ZnO$

Since making the above experiments, I have found that Hugo Tamm (*Chem. News* XXIV, p. 148) has proposed to weigh zinc as the double phosphate of zinc and ammonia,  $ZnNH_4PO_4$ , which, of course, necessitates the use of a weighed filter. His method is as follows: Make acid solution of a zinc salt alkaline with  $NH_4OH$ , then barely acid with  $HCl$  and add a solution of  $Na_2HPO_4$ , heat nearly to boiling for a few minutes, filter, wash, dry at  $100^\circ C$ . and weigh as  $ZnNH_4PO_4$ .

He states that there is invariably a loss when the double phosphate is converted into pyrophosphate by ignition; but from the results of my own experiments, I should say the loss was due to his precipitate being formed in an acid solution, in which I have found the double phosphate to be quite soluble. The greater quantity of zinc found when weighing the double phosphate was probably due to insufficient heat in the drying. He uses the atomic weight of 65.5 for zinc; using 65, the one now generally accepted, his results would all be low.

The composition of pyrophosphate of zinc is as follows :

2 Zn	130	42.763 per cent.
2 P	62	20.395 “
7 O	112	36.842 “
<hr/> Zn <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	<hr/> 304	<hr/> 100.000

or,

2 ZnO	162	53.29 per cent.
P <sub>2</sub> O <sub>5</sub>	142	46.71 “
<hr/> Zn <sub>2</sub> P <sub>2</sub> O <sub>7</sub>	<hr/> 304	<hr/> 100.00

Nickel and cobalt form phosphates which behave precisely like the corresponding zinc salt ; both, however, are strongly colored, and so can readily be distinguished from phosphate of zinc.

## VIII. METHYL VIOLET TEST PAPER.

By T.O'CONNOR SLOANE A. M., Ph. D.

Several years have passed since it was first proposed to use methyl violet as an indicator or test for mineral acids in the presence of organic acids. Since it has also been used as a coloring matter for test paper it occurred to me that a few notes on the preparation and use of the paper would be of interest.

I tried solutions of methyl violet of different strength in dyeing paper. A solution of one part of violet in four thousand parts of water was found to be sufficiently strong. A solution of double this strength gave a very good color.

Using paper dyed with the first solution I performed the following experiments:

One cc. of concentrated hydrochloric acid was mixed with him one hundred cc. of water. The acid was of sp. gr. 1.197 corresponding to about 40 per cent. acid; the solution therefore was of four one thousands strength or four-tenths of one per cent.

A piece of paper dipped in this solution did not show the reaction except on drying ; then it came out perfectly. A piece made from a violet solution of half the strength showed a little; this acid seemed therefore to be close upon the limits of sensibility for an immediate reaction.

It will also be noticed that the more weakly colored paper was slightly more sensitive ; on drying the darker paper shows as well or better.

The acid was diluted to one-fifth its former strength or eight-hundredths of one per cent. I now adopted the following way of operating: A convex cover was placed on a water bath, the water in which was heated to boiling; each piece of paper as dipped was laid on the upper convex surface of the cover; this dried it in about one minute; in practice this will be found an excellent way of using the paper.

The standard paper dipped once and dried showed the reaction, but not strongly; a second dipping and drying brought it out perfectly; a third treatment made the reaction still stronger.

When a paper colored by this weak acid was dipped a second time the previous change of color was indiscernible until it had dried again.

The acid was reduced to four-hundredths of one per cent.; the paper just showed the reaction after one dipping and drying; a second treatment developed it perfectly.

The acid was next reduced to two hundredths of one per cent. strength; the reaction appeared after three to five treatments but very faintly.

About the same results were obtained with sulphuric acid of corresponding strengths.

The series of experiments indicates pretty closely the limits of sensitiveness; drying the paper cannot be too strongly insisted upon; independent of any concentration of acid thus effected, the color change which is masked to a great extent by moisture is made visible by drying.

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## IX. DIPHENYLAMINE—ACROLEIN.

BY ALBERT R. LEEDS.

25 grms. of diphenylamine, in alcoholic solution, were treated with acrolein in excess, and after standing, the loosely-corked flask was gently warmed for a number of hours, until the smell of acrolein had nearly disappeared. A bulky dark red precipitate was formed. On boiling with alcohol a deep red solution was obtained and the portion undissolved formed a tenacious sticky mass, very awkward to work with. By repeated boiling with water under a return cooler this mass gradually lost its sticky nature. It was then digested alternately with boiling water and alcohol, until at last the mass became pulverulent and could be ground up in a mortar. The operation of boiling was then repeated many times, the



mass being powdered after each treatment with water, until at length the substance in a state of purity was obtained. Its analysis showed it to be Diphenylamine—Acrolein or, as it might be termed Didiphenylamineallyline,  $(C_{12}H_{10}N_2)C_3H_4$ .

	Found	Theory
Carbon,	86.26	86.18
Hydrogen,	6.29	6.36
Nitrogen,	7.28	7.45

It does not melt or sublime but is decomposed on breaking, leaving behind a carbonaceous residue extremely difficult of combustion. It is very slightly soluble in alcohol, insoluble in ether, and readily soluble in chloroform to a dark red liquid. From this solution, and also from that in glacial acid, in which it dissolves to a red liquid, but less readily than in chloroform, it could not be made to crystallize.

Its solution in chloroform was attacked with great energy by bromine. So also its solution in acetic acid, a dark red compound being formed on the addition of two atoms of bromide to one molecule of the diphenylamine. This compound was readily soluble in chloroform, but as it did not separate in a crystalline condition its analysis was not made. It was probably the addition compound  $(C_{12}H_{10}N_2)C_3H_4Br_2$ .

Its solution in acetic acid was attacked by nitric acid, forming a precipitate with a supernatant yellow liquid. Neither the solution or the precipitate yielded a crystallizable nitro-product, and their study was abandoned.

# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*April 7th, 1882.*

*Room 1, University Building.*

The meeting was called to order at 8.30 P. M.

Prof. Chas. A. Doremus in the chair.

The minutes of the previous meeting were read and approved.

The minutes of the Board of Directors were then read and accepted.

Mr. Elliott then moved that the regular order of business be suspended, in order to give more time for the election of the new committee on Papers and Publications.

Carried.

The Recording Secretary then stated that Messrs. Waller, Doremus and Friedburg had consented to serve on said committee, and he presented their names to be acted on.

After balloting, the above gentlemen were declared elected.

The recommendation of the Board of Directors that the Committee on Papers and Publications be authorized to issue a monthly number of the journal containing as much matter as the funds on hand will warrant, was carried unanimously.

After which the following gentlemen were elected as regular members of the Society.

Dr. G. Krause—Proposed by Geo. A. Prochazka, A. R. Leeds and James H. Stebbins, Jr.

Thos. S. Gladding—Proposed by Chas. M. Stillwell, T. J. Parker, and A. R. Leeds.

Prof. A. A. Brenneman—Proposed by A. R. Leeds, James H. Stebbins, Jr., and A. H. Elliott.

Geo. E. Perkins—Proposed by T. O'C. Sloane, M. Benjamin and W. Rupp.

Robert G. Remsen, Jr., M. D.—Proposed by A. R. Leeds, James H. Stebbins, Jr., and A. H. Elliott.

The following nomination was made:

G. E. Bailey as regular member—Proposed by M. Benjamin, W. Rupp and A. H. Elliott after which the first paper of the evening "On a new Volumetric process for Copper," by Mr. J. Casamajor was read.

This process consisted in precipitating the copper with an alkaline sulphide solution. The precipitate conglomerates, leav-

ing a clear liquid above so that by letting the alkaline sulphide run in slowly the point may be observed where no further precipitate is formed.

After some remarks by the chair, the second paper, "Upon the determination of tannic acid," by Mr. Nelson H. Darton then followed.

Dr. Grothe then stated that he together with Dr. Friedburg had recently observed the formation of a chloroform hydrate.

Some remarks and questions by the Recording Secretary then followed.

Dr. Friedburg then moved that members should be instructed to send all their papers and manuscripts to the Committee on Papers and Publications for their approval before they are read.

Carried unanimously.

Mr. Casamajor then presented the resignation of Mr. W. Schrage which was accepted.

Mr. Casamajor stated that Prof. Mallett had accepted an invitation for our coming annual dinner and moved that a committee of three be appointed to make arrangements for said dinner.

Carried.

The Recording Secretary then moved that the Chair appoint said committee.

Carried.

The Chair, after due consideration, appointed Messrs. Casamajor, Sloane and Stebbins.

Mr. Casamajor further stated that the cost for the dinner would be \$3 per plate.

After which the meeting adjourned.

JAMES H. STEBBINS, JR.,  
*Recording Secretary.*

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## X. VOLUMETRIC ESTIMATION OF COPPER AND OF LEAD

BY P. CASAMAJOR.

In this process, copper is precipitated from an alkaline solution by a titrated solution of sodic sulphide. This reagent is used in a process published some thirty years ago by Pelouze, in the *Annales de Chimie et de Physique*. In Pelouze's process, copper is dissolved in a large excess of ammonia, and an intensely blue liquid is obtained. To this a titrated solution of sodic sulphide is added until the blue color disappears.

To titrate the sodic sulphide, one gramme of pure copper is dissolved in nitric acid, and 40 or 50 cubic centimetres of concentrated ammonia are added, the whole being placed in a flask. This liquid is heated to boiling, and the solution of sodic sulphide is dropped in the flask until the blue color disappears. To observe this, the precipitate is allowed to settle, and more ammonia is added if necessary.

The solution of sodic sulphide was found to keep well in tightly stopped bottles.

I have had occasion to use this process several times, and have always found that the end of the reaction is not definite. The brown color of the sulphide, of which particles remain suspended, interferes with the observation of the color of the liquid. After the blue color has disappeared, an addition of alkaline sulphide will show a visible turbidity, even when copper is the only metal present.

Instead of treating a cupric solution by an excess of ammonia, I use an alkaline tartrate, dissolved in an excess of caustic soda. This liquid is the same which, added to a titrated solution of cupric sulphate, forms Fehling's solution. It is prepared by dissolving 173 grammes of Rochelle Salt in 480 cc. of caustic soda solution, of specific gravity 1.14, and adding water sufficiently to form 1 litre of liquid. This solution is added to the acid solution of copper to be tested, in slight excess of the quantity sufficient to redissolve the cupric oxide, and to obtain a clear blue solution. The liquid in the porcelain dish is then heated to nearly boiling point, and the sodic sulphide solution is gradually added, until no turbidity is produced by the addition of one drop of the reagent.

In this manner of proceeding, the blue color of the solution is not taken into account. The brown cloud which follows the addition of the titrated reagent is the only guide. The first portion of sodic sulphide, added to the alkaline cupric solution, gives rise to an intense black brown precipitate. As soon as this is formed the liquid in the porcelain dish is thoroughly stirred with a glass rod, which has the effect of agglomerating the cupric sulphide into a coarse curdy precipitate, which rapidly settles to the bottom of the dish, leaving the liquid quite clear and nearly colorless. If, after settling, the liquid should not be sufficiently clear, it should be vigorously stirred again, until the desired effect is obtained. After every addition of sulphide of sodium, the thorough agitation of the liquid is repeated until it is sufficiently clear. The degree of turbidity caused

by the addition of the reagent is a guide as to the quantity to be added. At the beginning, the brown color is very intense, and it is useless to wait for a complete clearing up before adding more of the reagent. Towards the end, the brown cloud is very slight, and the sodic sulphide should be added slowly, stirring up after every addition. Finally the sodic sulphide should be added drop by drop, until not the faintest cloud is seen after adding one drop.

By thoroughly stirring the liquid in the porcelain dish, and thereby causing the precipitate to become agglomerated in heavy clots, the surface of the dish remains very clean. On the white porcelain, as a back ground, the faintest cloudiness is easily observed, and a liquid containing one gramme of copper in 30,000 cubic centimeters, will still give a distinct brown cloud by the addition of one drop of the reagent.

For the volumetric estimation of lead, the same process may be applied. Sulphate of lead is easily dissolved in the alkaline tartrate solution. The sulphide of lead is precipitated in exactly the same manner as sulphide of copper. The precipitate agglomerates into curd as easily as does cupric sulphide, and the end of the reaction is equally definite.

As lead can be separated from other metals as sulphate, which is easily dissolved in the alkaline tartrate solution, we have by this method a rapid way of estimating lead. The precipitation by sodic sulphide is a quick operation, which only takes a few minutes. Sulphuric acid can also be separated as plumbic sulphate and estimated in the same way. Other acids which form insoluble plumbic salts could also be estimated by this process.

Copper can be separated from every other metal as sulphocyanate (Rivot's process). This precipitate may be heated with excess of nitric acid, and the alkaline tartrate solution added, after which the copper can be precipitated as sulphide.

These volumetric processes for copper and lead are founded on the easy agglomeration into heavy clots of the sulphides of these metals by agitation, the liquid remaining perfectly clear. Other metals, whose sulphides agglomerate in the same way in the alkaline tartrate solution, can probably be estimated by this process.

An attempt was made with silver, but chloride of silver was found to be insoluble in the alkaline tartrate solution. Chloride of silver was dissolved in excess of ammonia, and sodic sulphide added, but this did not prove satisfactory as sulphide of silver is sufficiently

soluble in ammonia to give a brown solution in which a faint cloud cannot be seen.

As chloride of silver is agglomerated into heavy clots by agitation, like cupric and plumbic sulphides, the white-cloud obtained, by pouring a titrated solution of sodic chloride into a solution of nitrate of silver, may be observed against a black background. This may be had by carrying on the precipitation in a flat dish of black glass, or in one of clear white glass, the under side of which has been covered with lamp black. The solution never clears up so completely by agitation as the alkaline solutions of copper and lead, but the slight milkiness of the liquid does interfere with the observation of the white cloud due to the formation of even very small quantities of chloride of silver.

The best temperature for the agglomeration of chloride of silver is about 65° C.

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## FOREIGN PATENTS.

Condensed from R. Biedermann's Report to the German Chemical Society, by Otto H. Krause.

A. R. PECHINEY, Salindres : *Manufacture of sodium bicarbonate* (Eng. P. 2098, May 24, 1880). This invention relates to the manufacture of soda by the ammonia process. The apparatus employed consists of a horizontal cylinder, which can be turned upon its long axis, and which is divided, first, by a perforated diaphragm parallel to the latter, and then into several compartments by discs placed at right angles to this. The discs have openings in the centre. The axles upon which the cylinder turns are hollow, to allow the gases and liquids to enter and pass out. Ammoniacal solution of sodium chloride enters, and unabsorbed gases pass out through the axle at one end, whilst the axle at the other end, which has two concentric openings, serves for the exit of the liquid containing the suspended sodium bicarbonate and for the entry of the pure carbonic acid resulting from the calcination of the latter. Impure carbonic acid, from kilns or furnaces, enters the outer concentric opening, and is led by means of pipes through the first three compartments of the cylinder and delivered into the fourth. The cylinder makes half revolutions at regular intervals. Ammoniacal solution of sodium chloride fills the apparatus, flowing through the holes in the discs, and passes into it continuously, whilst carbonic acid enters at the other end. On its way through, the liquid meets a current of gas, which latter gradually becomes richer in carbonic acid, the last three compartments receiving the pure gas.

R. I. T., N. T. and H. L. HAWKSLY, Oldham: *Forming salt into balls*, (Eng. P. 3789, Sept. 18, 1880). Salt intended for the manufacture of soda is formed into balls by means of a press, which also delivers them automatically upon the bottom of the furnace without breaking them.

W. THOMPSON, London: *Manufacture of white lead*, (Eng. P. 4056, Oct. 6, 1880.) Lead is placed in a chamber, the temperature of which is kept at 27—50° C. Vessels containing dilute acetic acid are placed on the floor. Carbonic acid and air enter through pipes placed above to complete the reaction.

WILHELM KAYSER, Ilmena: *Material for preventing boiler incrustation*, (Germ. P. 14189, Sept. 26, 1880.) Corkwood is pulverized and impregnated with barium chloride, sodium carbonate, or other substance suited to the water to which it is to be applied. The material so prepared pervades the water evenly, and gradually deposits after receiving the precipitate upon itself.

H. KOLBE, Hamburg: *Mercurial thermometer with electric alarm and variable contact*, (Germ. P. 13166, June 4, 1880.)

T. B. LIGHTFOOT, Dartford: *Cooling apparatus*, (Eng. P. 4065, Oct. 6, 1880.) An air ice machine in which the compressed and cooled air is expanded in two separate stages. In the first stage it is only expanded to such a degree as to deposit the greater part of its moisture. In the second stage it is then further expanded.

A. R. PECHINEY, Salindres: *Calcination of sodium bicarbonate in the ammonia soda process*, (Eng. P. 2099, May 24, 1880.) The invention consists essentially in heating the bicarbonate by means of heated carbonic acid resulting from a previous operation. This is done in four chambers of masonry enclosed in an iron casing which is pivoted upon the piston of a hydraulic press so that the whole can be lifted several inches. The heated carbonic acid gas is passed into the first compartment through the bottom, and heats the sodium bicarbonate placed on a grate. Through flues in the masonry the gas then passes in the same way through the second and third compartments, whilst the fourth is being discharged and refilled. When the contents of the first chamber has been sufficiently heated the apparatus is lifted and a quarter revolution given to it. The second chamber is then put into direct communication with the heated carbonic acid, etc. The carbonic acid evolved from the apparatus is passed through condensers to eliminate the water and then into holders to be used over again.

A. R. PECHINEY, Salindres: *Separation of liquids from precipitates*, (Eng. P. 2100, May 24, 1880). Consists of a filtering cylinder for separating the liquid from precipitated sodium bicarbonate by means of hydraulic pressure.



W., T., and J. CHADWICK, Manchester, and I. W. KYNASTON, St. Helens: *Separation of iron from aluminium sulphate, &c* (Germ. P. 14185, Aug. 31, 1880; addition to G. P. 11137; see this Journal, vol. II, p. 440, and vol. III, p. 72). To separate the iron completely the inventors add calcium ferrocyanide. To facilitate the settling of the Prussian blue formed, small quantities of a metallic salt are added. Preference is given to cupric sulphate, as any traces of it which may have remained in the solution can subsequently be removed together with the arsenic with hydrogen sulphide.

F. G. SPONNAGEL, Berlin: *Improvements in the manufacture of silicate of soda*, (Germ. P. 14182, Aug. 10, 1880). Precipitation from a concentrated solution by means of ether in a hermetically closed vessel.

W. CONRAD, Wurzburg: *Method of preparing blue coloring matters from the sulphonic acids which are formed by the action of ammonium sulphite upon nitroso derivatives of tertiary aromatic monamines*, (Germ. P. 14014, Jan. 20, 1880). By allowing ammonium sulphite to react upon nitrosodimethylaniline, the sulphonic acid of the reduced base is generated. By treating the latter with hydrogen sulphide and ferric chloride (Lauth, Ber. dent. chem. Gesellsch. 1866, p. 1035) a blue coloring matter is produced.

C. TIMMERMAN, Braunschweig, and R. GRAETZEL, Hanover: *Method and apparatus for the preparation of eurittonic acid from wood tar*, (Germ. P. 13787, Nov. 12, 1880; addition to Germ. P. 9328, see this Journal, vol. II, p. 301). Differs from the original patent chiefly in the manner of oxidizing the precipitated redissolved and dried dimethyl ethers of pyrogallol and methylpyrogallol acids. The air being passed over the powder heated to 160—220° C., instead of passing it through its solution in alkali.

A. DOMEYER and J. MARZELL, London: *Preparation of pure artificial alizarine*, (Eng. P. 2784, July 7, 1880). The melt from the anthraquinone-sulphonic acid is treated with water and the alkali neutralized with sulphurous acid. After separating the precipitated alizarine, the filtrate is rendered caustic by boiling with lime and the sulphurous acid recovered as calcium sulphite.

E. JACOBSEN, BERLIN: *Method of preparing benzoic acid, benzoic ether and benzaldehyde together with organic acid chlorides, acid anhydrides, &c., from benzo tri-chloride or dichloride in presence of certain metals, metallic oxides and salts*, (Germ. P. 13128, May 25, 1880; addition to Germ. P. 11494, see this Journal, vol. II, p. 462). The oxides, sulphides, hydroxides or other salts of the metals mentioned in the original patent can also be used in the processes there described.

E. PORION and L. MEHAY, Wardreques, France: *Manufacture of Alcohol*, (Eng. P. 2736, July 3, 1880). The raw material is sac-



charified by means of hydrochloric acid. In order to increase the value of the residues as fodder, the acid is freed from sulphuric acid by means of barium chloride. The sodium carbonate subsequently used to neutralize the acid must also be free from sulphate.

G. WASSMUS Berlin: *Manufacture of Alcohol from potatoes*, (Germ. P. 13637, Oct. 17, 1880). The saccharification is produced by means of hydrochloric acid, under a pressure of three atmospheres. The liquid is neutralized with sodium carbonate to render the residues suitable for fodder.

A. WUNSCH, Egel: *Preparation of Acetic Acid*, (Germ. P. 13165, May 11, 1880). The vapors containing acetic acid are passed through a rectifying apparatus, connected with a return condenser. Traces of acetic acid in the vapors leaving the apparatus are absorbed by some basic substance.

C. G. BJÖERKMANN, London: *Explosive Compound*, (Eng. P. 2483, June 19, 1880). Glycerine is mixed with one third of its weight of a carbohydrate, such as glucose &c. and then nitrated. The liquid is then mixed with combustible bodies containing oxygen, in such proportions that a powder results. It is said not to freeze and to be transportable without danger.

S. J. MACKIE, London: *Explosive compound*, (Eng. P. 4230, Oct. 18, 1880). Dry sodium nitrate is ground in a mill together with loose gun cotton. The mass is pressed in moulds, a split tube being inserted, which subsequently receives the fulminate.

JOHN PEARSON. J. WHITESTONE and R. R. GUBBINS, London: *Manufacture of Emery Wheels*, (Germ. P. 12924, May 11, 1880). A cement composed of glue, tannin and methylic alcohol is mixed with powdered emery, at 105° C. and compressed in moulds.

S. JAROSLAWSKI, Berlin: *Apparatus for carrying off the foul vapors from boiling animal matters*. (Germ. P. 13426, July 29, 1880.)—A pipe from the boiler ends in a nozzle like that of an injector and within a socket connected with a supply of water. The escape of the vapors under pressure aspirates a large amount of water which condenses and absorbs the bad odors.

R. C. ANDERSON, London: *Galvanic Battery*, (Eng. P. 4267, Oct. 20, 1880.)—Composed of carbon and zinc elements. The latter are immersed in solution of magnesium chloride, the latter in potassium chloro-chromate.

H. GROUVEN, Leipsic: *Method and apparatus for manufacturing ammonium sulphate from the nitrogen contained in peat*, (Germ. P. 13718, July 1, 1880. Addition to Germ. P. 2709.)—The retorts are placed horizontally instead of vertically, as described in the original patent. Peat, mixed with lime, is heated in a series of retorts which are so connected with each other that the vapors and gases from the first one are obliged to pass through the others.

The ammonium carbonate obtained from the cooled and condensed gases is converted into sulphate by digestion with calcium sulphate. The gases from the retorts after being deprived of ammonium carbonate and carbonic acid are burned in the retort furnace.

BINDSCHEDLER & BUSCH, Basle : *Method of preparing coloring matters by the action of halogens upon the azo-derivatives of resorcin*, (Germ. P. 14622, Dec. 30, 1880.)—The azo-derivatives of resorcin are obtained by the direct azotation of resorcin by means of a saturated solution of nitrous acid in sulphuric acid or by the action of nitroso compounds of phenols upon resorcin. Bromine (etc.) derivatives are prepared from the azo-compounds in alkaline solution. Upon acidifying the coloring substance is separated and can then be converted into the potassium or sodium salt. The bromine product forms a violet-blue to blue coloring matter with beautiful fluorescence.

BADISCHE ANILIN, UND SODA FABRIK, Ludwigshafen, am R. : *Method for the conversion of naphthols into their corresponding primary, secondary and tertiary monamines*, (Germ. P. 14612, Feb'y 22, 1880.) By the direct action of ammonia or of substituted ammonias upon alpha and beta-naphthol, alpha and beta-naphthylamine or their substitution products are formed. Of practical importance is the method for the preparation of the beta compounds, which hitherto could not be prepared on an industrial scale. For this reason also the azo-dyes of beta-naphthylamine and its derivatives have remained unknown until now. Two processes for the preparation of beta-naphthylamine from beta-naphthol are given.

W. MAJERT, Elberfeld : *Method of preparing a blue coloring matter*, (Germ. P. 14581, Aug. 3, 1880.)—If nitrosodimethylaniline in hydrochloric acid solution be allowed to react upon sulphocarbonic acid, the following reaction takes place :  $3 \text{C}_6\text{H}_4(\text{N O})\text{N}(\text{CH}_3)_2 + \text{CS}_3\text{H}_2 = 3 \text{C}_6\text{H}_4\text{N S N}(\text{CH}_3)_2 + \text{CO}_2 + \text{H}_2\text{O}$ .

On treating the solution of the resulting product, to which chloride of zinc and sodium chloride have been added, with an oxidizing agent, a blue coloring matter separates in flakes, whilst a red coloring matter containing sulphur remains in solution. The solution of the latter, reduced by means of zinc and hydrochloric acid until decoloration takes place, yields a further separation of the blue coloring matter upon addition of an oxidizing agent.

W. MICHAELIS, Berlin : *Method of testing cements and cement mortars*, (Germ. P. 13808, Oct. 5, 1880.)—The samples prepared in the ordinary way are subjected in steam-tight vessels to a temperature of 140° C. to 180° C. for twenty-four hours. This process shortens the time of setting and hardening of the cement considerably, so that the tests for strength, &c., can be applied immediately after taking the samples out of the apparatus and cooling them.

TH. RICHTERS, Breslau, and L. HAGEN, Magdeburg : *Purification of ammoniacal waste waters*, (Germ. P. 14210, Oct. 31, 1880.)—

The sewage, &c., is placed together with lime in closed vessels into which air is forced through a perforated pipe. The air, charged with ammonia, is passed through a Gay Lussac tower, where the ammonia is absorbed by sulphuric acid. The insoluble matters are allowed to subside in basins, passed through a filter-press, and then used for fertilizing.

L. MAUDIN and J. SCHNEIDER, Paris : *Method of removing bad smell and taste from alcohol*, (Germ. P. 13944, Nov. 9, 1880.)—Treatment with nascent hydrogen.

E. VAN HAECHT and J. SCHREINER, Drohobycz : *Method of obtaining paraffine from crude paraffine without the use of presses*, (Germ. P. 14507, Dec. 5, 1880.)—The oils are distilled off in a still by means of superheated steam until solid substances only pass over.

H. PRECHT, New Stassfurt : *Improvements in the method of separating sodium and magnesium chloride from potassium-magnesium sulphate obtained from Kainite*, (Germ. P. 13521, May 25, 1880. Addition to Germ. P. 10,637. See this Journal, Vol. II., p. 439.) A description of mechanical arrangements for separating the substances mentioned in the title as fast as they are formed, or shortly after the completion of the reaction.

L. WUESTENHAGEN, Hecklingen : *Method of and apparatus for evaporating the mother lyes in the manufacture of potash*, (Germ. P. 14015, May 1, 1881.)—The lyes, previously warmed, are run into a steam boiler for concentration, and finally evaporated in a vacuum pan to the point of crystallization. The steam from the boiler is superheated, and is used for heating the vacuum pan. The vapors from the latter are withdrawn by a pump and serve to warm the lyes.

SOCIÉTÉ ANONYME DES PRODUITS CHIMIQUES DU SUD-OUEST, Paris : *Improvements in apparatus for the manufacture of ammonia-soda*, (Germ. P. 14186, Aug. 31, 1880.)—Description of apparatus for absorbing the ammonium carbonate and ammonia evolved in the treatment of the ammonium chloride solution with limestone, dolomite and, finally, milk of lime.

C. DE MONTBLANC and L. GOULARD, Paris : *Improvements in apparatus for the manufacture of ammonia-soda*, (Germ. P. 14193, Oct. 2d, 1880; addition to Germ. P. 8498. See this Journal, vol. II., p. 106.)—Consist in providing the precipitating vessels with cooling jackets.

F. M. LYTE, London : *Method of obtaining lead and silver from mixed ores*, (Germ. P. 13792, Sept. 9, 1880.)—Relates to the treatment of mixed ores which cannot be easily separated on account of their nearly equal specific gravity, as galena and argentiferous lead sulphides, galena and calamine or zinc silicates, copper or iron

pyrites and zinc blende or galena, all or part of them mixed with barytes.

The general treatment consists in roasting the mixed ores to remove the greater part of the sulphur, (blendes, argentiferous lead glance and copper pyrites,) finely pulverizing and treating with hot hydrochloric acid, of 15—17%. Nearly all of the zinc and copper are thereby dissolved, whilst the greater part of the silver and lead remain undissolved in the gangue. The chlorides of silver and lead contained in the solution of zinc chloride precipitate upon cooling. By adding a fresh quantity of hydrochloric acid to the undissolved residue, nearly the whole of the lead and silver is dissolved. This solution is run into a second vessel containing a fresh quantity of the calcined ore where, by taking up zinc and copper, it becomes partially neutralized and the silver and lead chlorides are precipitated. By repeating this process the gangue, freed from zinc and copper, gradually becomes enriched in silver and lead chlorides. The latter are then extracted from the mixture by means of a hot concentrated solution of sodium chloride, from which the lead chloride separates upon cooling. The lead and silver in solution are precipitated with zinc and finally subjected to cupellation.

C. WINKLER, Freiberg: *Utilization of solutions containing metallic hyposulphates* ( $R^1S H O_2$ ) *or thio-sulphates* ( $R_2 S_2 O_2$ ) *formed by the sulphurous acid contained in acid gases and vapors*, (Germ. P. 14425, Oct. 14, 1880). Obtains ferric oxide, chloride and sulphate, sulphur and sulphurous acid.

N. VON LAVROFF, St. Petersburg: *Manufacture of Phosphor-lead-bronze*, (Germ. P. 14422, Jan. 23, 1881).

H. ERNST and A. FRICKE, Goerlitz: *Apparatus for the preparation of nitrogen from air by means of substances which absorb oxygen and with the aid of a pressure of several atmospheres*, (Germ. P. 13913, Mar. 2, 1880).

I. F. ESPENSCHIED, Friedrichsfeld. *Method of preparing violet, blue and green coloring matters by means of trichlormethylsulphochloride and use of the latter for the oxidation of leuco-compounds*, (Germ. P. 14621, Dec. 28, 1880). One part of methyldiphenylamine, and one to two parts trichlormethylsulphochloride are mixed with five parts sodium chloride and gradually heated to 110° C. A copper colored melt is obtained which is successively treated with water, alkali and concentrated hydrochloric acid to remove substances which remained unacted upon. The insoluble color-base is converted by means of sulphuric acid into a product soluble in water and treated further in the usual manner. This coloring matter dyes wool and silk greenish blue. Green and violet dyes are obtained by replacing the methyldiphenylamine by ethyl or amyldiphenylamine, or by benzyldiphenylamine, dibenzylphenylamine, diphenylamine or dimethylaniline.

For the oxidation of leucobases by trichlormethylsulphochloride

one part of the former is mixed with one part of tetramethyldiamidotriphenylmethane and five parts sodium chloride and heated to 110° C. The melt is boiled with water and treated with alkali.

C. ZIEGEL, Neuwedel. *Method of tanning hides*, (Germ. P. 13920, Aug. 24, 1880.)

I. & C. BALLATSHANO and H. TREUK, Berlin. *Method of preserving and waterproofing hides*, (Germ. P. 13420, May 8, 1880).

I. C. FELLNER, Frankfort of M. *Apparatus for driving gases out of liquids arranged for continuous working*, (Germ. P. 14204, Oct. 21, 1880).

L. PERRIER, Paris: *Vapor gauge for analyzing liquids and for ascertaining pressure*, (Germ. P. 13221, Aug. 21, 1880.)—This apparatus indicates the amount of alcohol contained in the vapors of stills. It consists of a pressure tube, the lower end of which is drawn out to a point which dips into the mercury contained in a small vessel soldered to the end of the tube. Above the mercury a liquid of known vapor tension is placed. From the height to which the vapor of this liquid forces the mercury in the tube the amount of alcohol in the distilling vapor may be judged.

C. R. ENGEL, Paris: *Method of preparing potassium carbonate*, (Germ. P. 15128, Jan. 29, 1881.)—Decomposes by heat potassium-magnesium carbonate, obtained by agitating magnesium carbonate with an aqueous solution of a potassium salt in presence of carbonic acid under pressure.

F. BENKER, Weinfelden: *Manufacture of sulphuric acid*, (Eng. P. 3540, Sept. 1, 1880.)—Injects the sulphurous acid into the lower part of the lead chambers in order to prevent waste of nitrate, &c.

H. BOECKEL, Stassfurt: *Method of working up Kainite and other double sulphates of potassium and magnesium with the aid of calcium chloride*, (Germ. P. 14938, Feby. 5, 1881).

C. SCHEIBLER, Berlin: *Preparation of magnesia from magnesian limestone, dolomite or calcareous magnesite*, (Germ. P. 14936, Jan. 1, 1881.)—The above mentioned substances are calcined to drive off carbonic acid, finely broken up and then treated with a 10 to 15 per cent. solution of sugar or syrup to dissolve out the lime. The solution of saccharate of lime is separated from the undissolved magnesia, &c., in a suitable manner and decomposed with carbonic acid to recover the sugar.

I. P. CLOSSON, Paris: *Improvements in the method of obtaining magnesia from calcined dolomite*, (Germ. P. 15342, Feby. 25, 1881, addition to Germ. P. 11456. See this Journal, vol. 3. p. 75.)—The decomposition of the solution of magnesium chloride by calcined

dolomite is greatly facilitated by the addition of small quantities of sugar or syrup.

B. E. R. NEWLANDS, London: *Manufacture of aluminium sulphate*, (Engl. P. 5287, Dec. 17, 1880).—Strongly presses the magma of impure aluminium sulphate to remove greater part of free sulphuric acid and soluble ferric salts.

C. HESSEL, Kilburn: *Preparation of hydrogen*, (Engl. P. 3584, Sept. 3, 1880).—A mixture of steam and hydrocarbons is injected into superheaters or retorts heated to redness. The carbonic acid formed is removed from the hydrogen by absorption.

F. X. BORSCHÉ, JR., Prague: *Improvements in the method of obtaining ammonia from the nitrogen contained in elution lyes, osmose waters, etc.*, (Germ. P. 14433, July 9, 1880).—The evaporated liquids are subjected to dry distillation over potash lime. The latter is obtained by moistening the carbonaceous residue of a previous operation, mixing it with slaked lime and heating.

F. J. BOLTON and J. A. WANKLYN, London: *Fertilizer*, (Engl. P. 5173, Dec. 10, 1880).—Acid calcium phosphate, extracted from commercial superphosphate, is mixed with ammonia water and the whole evaporated to dryness.

J. F. PARKER, Birmingham: *Manufacture of illuminating and heating gas*, (Engl. P. 3695, Sept. 10, 1880).—Hydrocarbons, tar oil or bitumen are mixed with 10–12 per cent. of lime, and heated in retorts. The gas is said to need no purification.

P. GREIFF, Frankfort: *Preparation of coloring matters of the rosaniline group by the action of nitrobenzyl chloride upon the salts of primary aromatic amines in the presence of oxidizing agents*, (Germ. P. 15120, Jan. 26, 1881).—A red, soluble in water, is obtained by heating to 170°–200° C. one molecule of nitrobenzyl chloride, two molecules of sulphate of aniline or toluidine and one molecule ferric chloride or other oxidizing agent.

FARBWERKE, successors to MEISTER LUCINS & BRUENING, Hoechst: *Method of preparing coloring matters from the nitro-derivatives of naphthalene*, (Germ. P. 14954, Dec. 17, 1880).—Monobrom-(Monochlor-) naphthalene is converted by energetic nitration into the tetra-nitrocompound



The halogen atom in the latter product can with facility be replaced by various molecular residues (hydroxyl, amidogen, etc.). From these various metallic salts of tetra-nitronaphthal are obtained which can be employed as coloring matters.

FR. GRASSLER, Cannstadt: *Diazotizing method and its continuation to the formation of coloring matters*, (Germ. P. 14950, Nov. 28, 1880).—This process differs from the one hitherto employed, in so far as no free acid is used for the decomposition of



the nitrite. The latter is employed in the form of ammonium salt or in combination with ammonium salts. The decomposition of the mixture is produced by warming the cloth to which it has been applied.

*Compagnie industrielle des procedes Raoul Pictet, Paris: Rectifying alcohol*, (Engl. P. 5245, Dec. 14, 1880.)—This method is based upon the fact that the difference in volatility between the empyreumatic substances, &c., and alcohol increases with the diminution of pressure. This method is said to yield alcohol of 99%.

F. THORN, Hamburg: *Extracting apparatus for analytical purposes*, (Germ. P. 14523, Oct. 17, 1880.)

J. STORER, Glasgow: *Apparatus for treating liquids with gases or vapors*. (Germ. P. 14900, Dec. 21, 1880.)—Within a vertical cylindrical vessel for holding the liquid a second cylinder, open at both ends, is placed axially and reaching to the surface of the liquid. A shaft studded with screw blades revolves at a rapid rate within the inner cylinder. By this means the liquid passes rapidly through it, air or other gases are aspirated into it, and the latter intimately mixed with the liquid.

PAUL CASAMAJOR and CH. H. SENFF, New York: *Improvements in filtering apparatus*, (Germ. P. 14937, Feb'y 1, 1881.) See under "American Patents," this Journal, vol. 3, p. 65.

H. BORNTREGER, Wurzburg: *Method of manufacturing sulphuric acid, free from arsenic and selenium, from the soda residues of Leblanc's process, with the aid of exhausted pyrites*, (Germ. P. 15757, Mar. 8, 1881.)—Sulphide of iron is reformed by digesting the finely powdered exhausted pyrites with the sulphur lyes, obtained by extracting soda waste with water under pressure,

TH. RICHTERS, Breslau: *Method of obtaining ammonia from the purifying mass of gas works*. (Germ. P. 15206, July 31, 1880). The oxide of iron mixture as it comes out of the purifiers contains about 0.8%  $\text{NH}_3$ , this amount is lost by volatilization during the process of regeneration of the mass as ordinarily conducted, i.e. exposure to the air &c. The inventor combines the recovery of this ammonia with the regeneration of the mass, by putting the latter into tanks provided with diaphragms and passing air through it. The heat resulting from the reaction drives off the ammonia which is then obtained from the exhaust air by treating the latter with acid.

A. & L. BRIN, Paris: *Oxygen from atmospheric air*. (Germ. P. 15298, Feb. 2, 1881). Baryta is heated in retorts, in the usual manner, first at a low temperature while passing air over it, then at a higher temperature to decompose the barium peroxide. The regulation of the temperature is accomplished automatically by means of pyrometers, connected with dampers which control the supply

of air admitted to the furnace. The oxygen is exhausted from the retorts by a barometer pump, consisting of two cylinders alternately filled with water which also work automatically.

A. BAEYER, Munich: *Method of preparing paranitrobenzaldehyde* (Germ. P. 15743, Feby. 20, 1881). This substance is obtained by the limited oxidation of paranitro-cinnamic acid, or of the ethers of this acid. For this purpose the following oxidizing agents are employed: potassium permanganate in alkaline and acid solution, lead peroxide, manganic oxide and sulphuric acid, chromic acid, potassium chromate and sulphuric acid, nitric acid, or a mixture of potassium nitrate or nitric acid, and concentrated sulphuric acid.

LEMBACH & SCHLEICHER, Biebrich am R.: *Method of preparing coloring matters by the action of nitrobenzylchloride or bromide upon secondary and tertiary amines or phenols* (Germ. P. 14945, Aug. 21, 1880). From these reactions which must take place in the presence of oxidizing metallic salts, blue, green, violet and red coloring matters result.

M. PRUD'HOMME, Mulhausen: *Method of preparing coloring matters for cotton, wool and silk* (Germ. P. 15616, Jan. 28, 1881).—From anthraquinone, alizarin, anthrapurpurin, flavopurpurin and Cœrulein nitro substitution compounds are prepared which by heating with sulphuric acid exchange the  $\text{NO}_2$  group for the  $\text{SO}_3\text{H}$  group. These sulphonic acids, their salts and finally the hydroxyl substitution products which result from the fusion of the sulphonic acids with alkaline hydroxides are the coloring matters patented.

E. A. SCHOTT, Kreiensen: *Manufacture of carbonized peat coal for removing coloring matters and impurities from liquids and for disinfecting atmospheric air in enclosed spaces*, (Germ P. 14923, Dec. 14, 1880).

B. J. MILLS, London *Explosive compounds* (Engl. P. 800 and 801, Feb. 24, 1881) 9295 parts nitroglycerine, 5–7 parts moderately nitrated cellulose and 0.5–2 pts. alcohol. Another compound is composed of 0.5 to 3 parts moderately nitrated cellulose, 23 to 30 parts sodium nitrate, 8 to 18 parts wood cellulose and 3 to 4 parts sulphur.

H. PROUDHOMME, Putte Capellen: *Explosive compound* (Engl. P. 4200, Apr. 14, 1881).

C. HEINZERLING, Frankfort of M *Rapid tanning process by the use of chromates or chromium salts in combination with other mineral substances and dissolved fats and with hydrocarbons*, (Germ. P. 14,769 Nov. 9, 1880). Relates to the manner of applying the solutions to the hides, the modifications which can be made in the composition of the solution to suit the uses to which the leather is to be applied, and the manner of fixing the absorbed matters, &c.



## XI. ON THE COMPARATIVE VALUE OF THE SEVERAL METHODS OF DETERMINING TANNIC ACID.

BY NELSON H. DARTON.

This investigation was to determine the relative accuracy of the following methods: Lœwenthal's latest modifications of his old method; Hammel's skin and specific gravity process; Carpené's indigo method as modified by Barbieri; the old gelatin process, highly refined; Allen's lead method, as modified by Sanford, and my own method, a modification of Lœwenthal's.

In the first place, I would like to state that every evidence that I can bring to bear shows that there is but one tannic acid—a principle which has the peculiar property of combining in the proportion of 10 parts by weight with 6 parts by weight of skin to form leather. In nature this principle is often mixed with glucosides, so-called extractive matter, etc., and hence the various formulæ assigned to what have been supposed to be different tannic acids. I propose to show proof of this in a subsequent paper. Where tannic acid is mentioned in this paper the above principle is meant. It can readily be obtained pure from nut galls and oak and hemlock bark. When obtained from kino and catechu (the so-called catechu tannin) it is accompanied by an extractive principle which renders the tannate of copper soluble in ammonia.

For the purpose of standardizing the numerous solutions to be used, an absolutely pure tannic acid was examined. Its preparation was conducted thus: A fair quantity of coarsely ground Aleppo nut-galls was taken and treated with ether and water, after the method of the U. S. P.

The product thus obtained was then thoroughly washed with absolute alcohol, dissolved in absolutely pure ether, and this repeatedly agitated with water until all of the tannic acid was thought to be extracted from the tannin; it was then separated from the ether by evaporation finally in a vacuum; was then dissolved in 80 % alcohol, filtered and separated from this as above; this well dried over sulphuric acid, and at a gentle heat in the air bath in a current of pure, dry carbon dioxide; a portion of it was burnt, and twice yielded a proper formula.

A solution of this was prepared containing 2 grams to a litre;

this solution was kept in a colored bottle out of contact with the air by carbon dioxide, and when not in use, for greater precaution, the bottles were placed in the dark. No change however occurred in their relation to the permanganate solution.

Pure gallic acid was prepared from nut galls. It was carefully recrystallized and readily obtained pure.

A solution of this was prepared as of the acid and was carefully kept.

Pure English gelatine (Nelson's), was used after purification, and a solution of it was prepared immediately before use by dissolving 25 grams in a litre of a 10% solution of sodium chloride.

The permanganate of potassium solution contained 1.5 grams to a litre, and was frequently renewed during the investigations, the acetate of lead solution for Allen's method contained a deci-nomal quantity of the pure acetate of lead.

The indigo carmine solution was prepared by dissolving 3 grams of the pure preparation in a litre of water. It was boiled for some time and carefully kept; did not change its relation to the permanganate solution a fraction of a cubic millimetre. The other solutions were all prepared as required with every care and precaution.

For the objects of the determinations, decoctions were prepared of hemlock, oak and chestnut barks, Aleppo nut galls, sumach leaves, and of the so-called catechu, tannins of catechu, kino and rhatany root. As they were all prepared and kept under the same conditions, I will only detail the mode of extraction of the first and most important one, hemlock. To a litre of the decoction in view 20 grams of the coarsely ground select inner bark of the hemlock was taken; this was extracted by carefully heating it with water in a current of carbon dioxide, allowing the decoction thus formed to cool, and then filtering it through four thicknesses of filtering paper; a very small amount of dilute sulphuric acid was then added to the solution and it was again filtered, the excess of acid neutralized by baryta water and filtered. The decoction was thus freed from the so-called extractive matter and contained tannic and gallic acids nearly pure. This decoction was preserved in an atmosphere of carbon dioxide. The decoctions of catechu, etc., were prepared and kept in a similar manner but still retained a strong color; they are, I may add, much less liable to change, owing to the peculiar principles present. Tannic acid prepared from these solutions by a process similar to the one used for galls, had a variable

constitution, differing materially from that of the pure tannic acid obtained from the others.

I will first consider Lœwenthal's method (Fres Zeitschr. 1877, pp. 33 and 201.) This method has been highly commended by Proctor and others, but in working with it I soon found that it gave results at variance with practical experience and with my control method as will be detailed later. Portions of each of the ten different decoctions were taken and the analysis conducted in strict accordance with Lœwenthal's directions. Thus : 20 cc. of the indigo solution was first titrated. It required exactly 8.4 cc. of permanganate solution. The titrations were conducted in a porcelain dish;  $\frac{3}{4}$  of a litre of solution and 10 cc. of dilute sulphuric acid were used in every case. 10 cc. of the standard tannin solution, with 20 cc. indigo solution and acid as above, required 13.7 cc. Deducting 8.4 cc. for the indigo we have 5.3 cc. used, for the tannin.

This same operation was repeated, using the decoction instead of the standard solution. 15.3 cc. were now used, or 6.9 cc. for the decoction.

100 cc. of the decoction were then taken, placed in a flask with 50 cc. of the gelatine solution and 100 cc. of a saturated sodium chloride solution, containing also 25 cc. of sulphuric acid and 50 cc. of hydrochloric acid per litre. Between the additions the mixture was well agitated. It was allowed to stand over night, and a perfectly clear filtrate taken off the next morning. To save calculation 25 cc. of this, equaling 10 cc. of the undiluted decoction, were taken and tested as above, using 11.45 cc. of permanganate, or 3.05 cc. for the impurities, etc., not tannic acid. Subtracting this from the figure 6.9 cc. leaves 3.85 cc. and we have the equation;

$5.3 : 3.85 = 0.2 \text{ grms. to a litre} : x \text{ grms. to a litre.}$   
 $x = 1.45 \text{ grms. to 20 grms. of bark, or 7.25 per cent., the value of the bark.}$  This, as I will show beyond, is lower than is really the case. The figures obtained were:

No.	20 G Hemlock.	20 G Oak.	20 G Chestnut.	15 G N. Galls.	10 G Sumach.	10 G 5 Catechu.	2.5 G Kino.	3 G Rhatany.
1	7.25	7.40	6.00	52.5	13.5	47.0	82.0	About 80.00
2	7.00	7.30	6.00	56.3	12.9	51.0	82.0	
3	7.30	7.45	6.10	54.2	12.9	48.0	82.0	
4	7.20	7.25	5.90	55.0	13.3	49.0	82.5	
5	7.25	7.00	6.30	55.5	13.1	48.0	81.3	
6	7.20	7.00	6.00	53.0	12.8	50.0	83.0	
7	6.9	7.16	5.90	55.6	13.2	51.0	82.0	
8	7.25	7.30	6.20	54.4	13.25	49.0	82.0	
9	7.10	7.20	6.00	54.5	12.95	50.0	83.0	
10	7.15	7.10	6.15	52.8	13.2	48.0	81.5	
Average.	7.16	7.21	6.15	54.4	13.1	49.0	82.1	34.00
Real Value.	7.90	8.22	7.42	61.5	16.25	40.0	72.0	

giving altogether an average value about 95 out of exactly 100 per cent. But as this is an average including the discordantly high and low it is not to be wondered at. The value however for the first five is about 86 per cent. and for the last two 117 per cent.

The errors are incurred because the tannate of gelatine is more or less soluble in a weak gelatine solution even when chloride of sodium or the strong acids are present; in fact although the acids facilitate coagulation they also dissolve a portion of it. It is also somewhat soluble in solutions of gallic acid as is shown by the figures in the nut gall column of the table; moreover much gelatine remains in the solution. The quantity is very variable as it depends upon the amount of so-called extractive matter present. As I will show further on all those errors together produce an effect—amount to perhaps 40 per cent. This is, however, partly complicated by the amount of oxidizable matter and gallic acid which are also, when together, precipitated partly with the tannate of gelatine by the withdrawal of the tannic acid which holds them in solution. This is shown in the column under rhatany where so much matter depending on the tannic acid for its solution was precipitated as to render even approximation impossible. It is also shown in catechu and kino but to a less extent; this is what some in good faith call tannic acid, and ascribe to it a formula. They probably never combined an ounce of tannin with skin in all their lives, so their ignorance is pardonable. In the last three determinations, I used as near as possible the exact amount of gelatine necessary and a minimum of acid solution, with but little difference in the results.

This method surely is a very satisfactory one as far as it goes, as it

is very easy to execute and has not so many of the difficulties peculiar to some of the others; it however, does not come up to my requirements. The value of this method, especially its use in determining the values of spent liquors was investigated. These liquors contain many unknown principles derived from the skins in their treatment, and thus their accurate determination is very difficult. This and another method of determination are the only ones that can be used at all. It is of great importance to the tanner to know how his spent liquors and the spent barks run in strength. The spent liquors were taken, treated with sulphuric acid, etc., as above, and the determination proceeded with. Five analyses were made of the spent liquors direct from the vats after a run with Buenos Ayres skins well cleaned and raised—oak and hemlock liquors and those from catechu were also taken. They had been used on five skins but were good samples. The results are stated in absolute per cents.

No.	Oak.	Hemlock.	Sumach.	Catechu.
1	.13 per cent	.08 per cent	2.95 per cent	2.90 per cent
2	.14 per cent	.085 per cent	2.90 per cent	2.93 per cent
3	.125 per cent	.08 per cent	2.95 per cent	2.93 per cent
4	.16 per cent	.08 per cent	2.93 per cent	2.89 per cent
5	.145 per cent	.085 per cent	2.90 per cent	2.90 per cent
Aver.	.140 per cent	.0825 per cent	2.93 per cent	2.91 per cent
Real value	.27 per cent	.21 per cent	1.62 per cent	1.90 per cent

These figures are, of course, only approximate, but are quite exact proportionally. The causes of such great errors are these: Much less matter is separated with the tannate of gelatine, a very large proportion in fact, nearly all of the latter dissolves and the amount of gelatin left in solution is very considerable, so that there is merely a perceptible difference in each two determinations. With spent barks there is but little difference from the result noticed above, they being a little higher by compensation.

Now the prime argument. To show the misleading results given by this method and to show the control used, we will consider hemlock bark No. 1 analysis all through: Here in the first table we find it to contain 7.25 per cent. of tannic acid. We take a cord of this bark, leach it, and obtain 1,250 gallons of decoction. The spent bark is analyzed by this method, and found to contain .7 per cent. of tannic acid. Leather is tanned from the decoction with great precaution, and what is 5 per cent. of the bark found to have been used. As I will show further on, this was pure

tannic acid. The spent liquor is also examined, and found to hold what was equivalent to 5 per cent. of the bark in tannic acid. That is to say, the same—as we would speak of the spent liquors as containing nearly all the tannin of the bark, or 6.5 or 7 per cent. of it, in tannic acid. However, all these figures added together give a result of 6.2 per cent., or 1 per cent. unaccounted for, thus showing the result to be too low. The tannic acid had not decomposed, as I used a preservative to keep it, and one which I know to be thorough and complete.

Now, if the spent liquors be examined by say the method I will note further on, we find it to contain just twice as much, or 1.4 per cent. This would bring it up to 6.9 per cent.; still too low; but by examining the spent bark in the same process we have 1.5, instead of 5, and the figure is brought up to its nominal value then proved of 7.9 per cent.

According to the reasoning deduced from this process, we would have 6 per cent. of the bark utilized as tannin to combine with the leather. Nothing is more erroneous than this, as is proved every day by the tanner; as never over 5 per cent. is obtained from hemlock, the others in close proportion. So much for this process.

Next in order of our consideration comes Hammond's method, and its modifications, which were so popular some time ago; among some of the tanners the results obtained by it were so inaccurate and inconsistent as to speedily set it aside. The many inaccuracies have been partially shown by a number of investigators, especially Proctor, who has shown that gallic acid is thrown down with the tannin, etc. The method was as carefully tested and examined as the other, and the results are here tabulated:

No.	Hemlock.	Oak.	Chestnut.	Nut Galls.	Sumach.	
1	10.2	13.2	10.0	34.0	10.00	Catechu, Kino, and Rhatany, not ac- cordant results.
2	6.5	6.4	4.8	43.0	11.00	
3	9.9	9.3	9.2	56.0	6.5	
4	11.4	12.4	7.4	40.0	7.6	
5	9.6	14.0	6.9	61.0	9.8	
6	6.8	4.0	11.2	52.0	6.9	
7	11.2	8.0	10.2	57.0	8.2	
8	6.9	12.5	4.9	49.5	7.4	
9	8.4	5.0	8.6	45.0	10.5	
10	10.5	10.0	9.4	56.0	6.8	
Average, Real value,	9.14 7.90	9.48 8.22	8.26 7.42	49.0 61.5	8.57 16.25	

These figures were obtained in the following manner. The decoctions, filtered and treated as before, were placed in a graduated jar, and the specific gravity taken by the use of a very delicate densimeter. The skin was then taken, thoroughly cleansed, heated and raised by appropriate treatment. It was then thoroughly washed with a solution of gallic acid, again with acetic acid and warm water, until it was pure and sweet. It was then placed in water with the decoction, and allowed to air for an indefinite period, for the first five analyses. It was then taken out and scrubbed off as far as possible without altering the bulk of the solution, the specific gravity of which was then taken. The amount of tannic acid was calculated from the difference, etc.

The details of the method were executed with much more refinement than is the case generally, and the principles involved thoroughly worked out. But with this look at the result one might reason, Why would not the result be identical with the practical manufacture of the leather, or 5 per cent, etc? This I can readily show. In the first place, to obtain the decoction for their determination, or rather to make solutions uniting all the tannin in the material used, we have to extract much more extraneous matter, especially in the case of rhatany, kino and catechu, than in the imperfect leaching of the tanneries, where in the case of the last materials only 50 per cent. of the tannin present, and a minimum of coloring matter, are extracted; thus the leather has less to contaminate it. In the second place the sample of skin is never a fair specimen in the treatment of a whole hide, and besides, the leather formed cannot be properly freed from the different matters besides tannic acid soaked into it, without altering the volume of the solution. It must be remembered that leather after it is formed is thoroughly rolled, and scrubbed so as to leave nothing but leather. The leather absorbs tannic acid and the so-called extractive matters; and by withdrawing the tannic and partly the gallic acid from a solution much matter is deposited, partly in the solution, some in the leather. This by the above treatment is removed before the leather is dried and weighed; thus much matter is abstracted and accounted for as tannic acid, this yielding too high results. Besides these considerations the densimeter must have extreme delicacy, and even then a small space on the scale represents much tannin. Altogether, I pronounce this method little better than a guess; in fact a tanner can arrive at the strength of his liquors by their taste better than by this method.

In the examination of spent liquors and barks this method was of no value at all, as it gives such very inconsistent results. This is caused by the putrescent matters present in the liquor and other causes. In the spent liquor the analyses turn out like those shown in the table below :

No.	Oak.	Hemlock.	Sumach.	Catechu.
1	6.5	4.0	7.9	4.0
2	2.2	5.2	8.4	3.0
Average,	4.3	4.8	8.2	3.5
Real value,	.27	.21	1.62	1.90

In fact, Hammel's whole method, however modified, is the same thing. This instrument should be done away with in the tanneries as it only shows how much matter, tannic and otherwise, especially otherwise, *is in solution*.

In the use of this method and the worthless modification of drying the skin filterings and weighing before and after treatment, chemists and others generally obtain the results too low, but that depends on the very nice point of understanding how to make leather.

Using the calculations applied to Læwenthal's process for a control we find thus: In No. 1 the leather 5 per cent., the spent liquor 3.5 per cent., the spent bark 4.5, all together 13 per cent. This is quite far from the average value of the bark, which will be noticed was 9.48. Giving the proper value to the spent liquor (1.4 per cent) the result is 10.9, or much nearer the truth.



# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

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Meeting held May 5th, 1882.

The meeting was called to order at 8:40 p. m. Prof. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

The report of the Board of Directors was read and accepted.

Dr. Doremus on behalf of the Committee on papers and publications stated that he was able to present to the society this evening, all the proceedings, and nearly all the papers read before the society from January 1st up to date.

He further stated that the next number of the Journal would be out by May 25th. He also said that the Committee are in need of more abstractors and earnestly hoped that the society would support them in this direction.

As there are from three to four fly leaves of the Journal that can be devoted to advertising, the Committee thought that the money that could be obtained from that source would be a great help to the Journal.

The present issue of the Journal would cost about ninety dollars, the number of pages being somewhat larger than was originally intended. But he thought that if the Committee could count upon publishing a number of the Journal of thirty-two pages each month, that the cost would be reduced to sixty dollars per month, or about five hundred and fifty dollars for the year, including the ninety dollars for the present month.

The treasurer stated that money was coming in fast at present.

The librarian had nothing to report except that a number of periodicals had come in.

The following gentlemen were nominated :

G. Polledo, as associate member. Proposed by T. O'C. Sloane, James H. Stebbins, Jr. and Arthur H. Elliott.

H. Edward Stockbridge, as regular member, by C. A. Goessmann, H. Endemann and James H. Stebbins, Jr.

Dr. Wm. Stratford, as regular member, by Charles A. Doremus, R. A. Witthaus and James H. Stebbins, Jr.

E. J. Mallett, Jr., as regular member, by Charles A. Doremus, James H. Stebbins, Jr. and A. R. Leeds.

After which the first paper of the evening, on Acrolein-urea, was read by Dr. A. R. Leeds. After some questions by Mr. Elliott, the second paper of the evening. Note on an earthy ferric sulphate, by Charles E. Wait followed. Mr. Darton's paper on the determination of tannic acid was then read by title.

The following papers were announced for the next meeting:

1st. Use of the tannometer in tan yards, by Nelson H. Darton.

2nd. On the determination of phosphorus in iron ore, by E. Waller.

3rd. Report on the progress of industrial chemistry, by Dr. E. E. Brenneman.

Dr. Waller moved that when the meeting adjourns, it shall be adjourned to the next conversazione. Seconded and carried.

Dr. Doremus moved that all papers read before the society shall become the lawful property of the society till thirty days from the date of reading. After a number of suggestions by different gentlemen, Dr. Doremus' motion was carried. The meeting then adjourned.

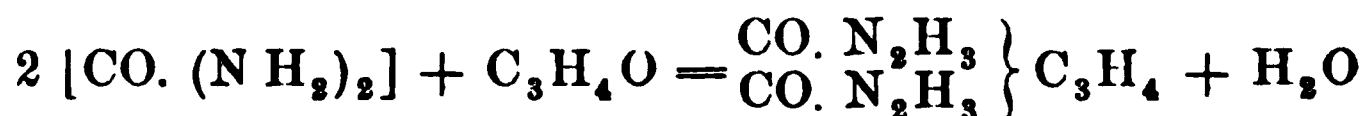
JAMES H. STEBBINS, Jr.,

*Recording Secretary.*

## XII. ACROLEIN-UREA.

BY ALBERT R. LEEDS.

In the *Annalen der Chemie und Pharmacie* for 1869, (vol. lxxv p. 203), Hugo Schiff has described, under the name of acrylureid, a condensation product formed by the union of two molecules of urea with one of acrolein, in accordance with the reaction:



Without being aware of Schiff's labors, and whilst studying the aldehyde compounds with quite different objects than those followed by Schiff, I obtained the same compound, but had assigned to it a quite different formula. The explanation of these differences becomes apparent on studying the method by which Schiff prepared the compound, since this method did not and could not yield the substance in question. He mixed a concentrated aqueous solution of urea with acrolein, and after his acrylureid had gradually precipitated as small white needles (?), he washed it merely with ether and water, and dried *in vacuo*. He likewise states that powdered

urea unites with acrolein directly. "The mixture swells up, and is converted into a white brittle mass, which in some places has an entirely crystalline appearance. By far the greatest portion is intimately mixed with a white porcelain-like product, formed by decomposition of the acrolein." The analysis of this body gave, as might readily have been expected, no satisfactory results. Schiff further states that in various preparations, some of which were prepared at ordinary and others at higher temperatures, he obtained carbon varying between 44 and 47 per cent., and hydrogen between 6.9 and 7.1 per cent. The formula of diacryltriureid requires 42.2 per cent. carbon and 6.3 per cent. hydrogen. That of acryldiureid, which Schiff adopted, but 38 per cent. carbon and 6.3 per cent. hydrogen.

Throughout his investigation Schiff worked upon impure substances, and in that way obtained not only varying results, but results which would support no rational formula, the one which he finally adopted,—that of diacryltriureid, being altogether erroneous.

In the first experiment made by the author, the acrolein obtained by heating anhydrous glycerin with potassic bisulphate was allowed to distil over directly into an alcoholic solution of urea. A precipitate formed at once, and after a time a considerable amount was obtained, the urea however being still in excess. The filtrate was of a reddish color, and on evaporation yielded a resinous or gummy mass, which could neither be sublimed or crystallized from solvents. The precipitate left on the filter was of a yellow-white color, but after repeated treatment with alcohol became quite white, and lost all of the gummy or thick oily substance at first adhering to it.

Its analysis showed that it had been formed by the union, not of two, but of one molecule of urea, with one of acrolein, a molecule of water being eliminated, and the resulting compound having the formula  $C_4H_6N_2O$  or  $CO(NH)_2C_3H_4$ .

	Found.	Theory.
Carbon	49.01	48.98
Hydrogen	6.3	6.12

Not only as originally prepared by repeated purification with alcohol, but also after treatment with carbon disulphide, chloroform and ether, in all of which it is only slightly soluble, every attempt to obtain it in a crystallized condition was unsuccessful. It did not melt nor sublime, and when strongly heated underwent decom-

position. It was readily attracted by nitric acid, but the products of the decomposition were not studied. From its solution in alcohol no precipitate was obtained on the addition of bromine. On treatment with hydrochloric, tartaric and acetic acids no salts were formed.

The alcoholic extracts were collected, and yielded, after successive solutions in alcohol and precipitation with water, a white substance similar to that above described, and having a melting-point of  $185^{\circ}$ . Since sufficient of this material could not be obtained to admit of its satisfactory purification and analysis, a much larger amount of urea in alcoholic solution than that used in the foregoing experiments (40 grms.) was treated with acrolein in successive portions in a closed vessel, until after long continued warming the odor of acrolein ceased to disappear. In this case, unlike the foregoing, no precipitate was formed, nor did any take place until after the addition of water, when a very abundant white amorphous substance was thrown down. This was boiled with alcohol a great many times, each extract affording a white precipitate when diluted with water, and the portion of pure material obtained eventually was not more than that procured in the first instance, when but a small amount of urea was used and the acrolein was not added in excess. The composition of the substance thus obtained was  $\text{CO. (NH)}_2\text{. C}_3\text{H}_4$ , and it was identical in all respects with the substance formed in the first case. From the alcoholic extracts no other substance could be obtained, and the explanation of the small yield of the pure acrolein-urea in the second case, was eventually found to be due to the presence of the oily or gummy substance, formed as a by-product. The amount of this by-product was apparently much greater in the second case, and explained the non-precipitation of the acrolein-urea until after the addition of water to the original solution of the urea in alcohol. And the tenacity with which it adhered to the acrolein-urea on precipitation with water, and carried it into solution again on each successive boiling with alcohol, explains the very small eventual yield of the pure material. It was also due to the small adherent traces of this oil, that the second body, the one soluble in alcohol, had a melting point of  $185^{\circ}$ , and was supposed to be something else than the acrolein-urea.

The presence of this oil, which I was unable to isolate, explains all the above difficulties and the erroneous results obtained by Schiff. In conclusion, it may be safely stated that besides the small amount of this oily by-product, the only compound formed by the

action of acrolein on urea, was that containing the residue from one molecule of urea, the compound  $\text{CO}(\text{NH})_2 \cdot \text{C}_3\text{H}_4$ .

### XIII. NOTE ON AN EARTHY FERRIC SULPHATE FROM ARKANSAS.

BY CHAS. E. WATT.

Several weeks ago a sample of earth was sent to me for examination, from Southern Arkansas. It is a friable, yellowish substance, partly soluble in water, streak uncolored, taste astringent.

Upon analysis I find it contains the following :

	PER CENT.
Insoluble in cold water.....	12.61
Soluble in cold water.....	87.39
	<hr/>
	100.00

The soluble portion consists of

$\text{Fe}_2 \text{O}_3$ .....	21.82
$\text{SO}_3$ .....	33.81
$\text{H}_2\text{O}$ .....	31.76
	<hr/>
	87.39

By assuming the following arrangement,

$\text{Fe}_2 \text{O}_3$ .....	21.82
$\text{SO}_3$ .....	33.81
$\text{H}_2\text{O}$ combined.....	22.41
$\text{H}_2 \text{O}$ hygroscopic.....	9.35
	<hr/>
	87.39

we are able to deduce a formula consistent with that of the native persulphate of iron, viz.:  $\text{Fe}_2 (\text{SO}_4)_3 (\text{H}_2\text{O})_9$ .

If all the water be considered as belonging to the compound, the formula will be  $\text{Fe}_2 (\text{SO}_4)_3 (\text{H}_2\text{O})_{13}$ .

It is said an unlimited supply of this substance may be obtained.

#### XIV. ON THE COMPARATIVE VALUE OF THE SEVERAL METHODS OF DETERMINING TANNIC ACID.—PART 2.

BY NELSON H. DARTON.

The next method examined was that of Carpené, as improved and modified by Barbieri. This method was looked forward to as an excellent one when first proposed, and consequently was brought into use almost immediately. The details of the execution were very simple, and its originators obtained excellent results. Some time after, Kathreiner examined into its value, and stated\* that with it he had only been able to obtain exceedingly inaccurate and discordant results, and had failed in his endeavor to favorably modify it. In his investigations he employed a decoction of sumach and a solution of the 'so-called "pure tannin" of the shops, and thus the comparison between the sumach and this compound, containing not over 80 per cent. of digallic acid, was not a correct one. The other 20 per cent. of the so-called "tannin" was composed, as I have found, of matter nearly all precipitable by ammonia solution, and the importance of taking this into consideration is shown below. Thus the poor results obtained by Kathreiner are explained. I have, in the investigation of this subject, applied this method to most of the other tanning materials, and have in this manner brought some new facts in relation to it to light, not only by employing the variety of the substances tested, but also by varying the conditions of procedure and investigating the source of the errors. My general mode of procedure was as follows: Using as before, for example, the decoction of hemlock bark, though not in strict accordance with the details as published by Bolley in his *Handbuch*, but with as much modification as experience had shown to be desirable. The decoction was taken in convenient quantity, an excess of a solution of ammonia added to it, and the precipitate thus produced filtered out. A known amount, which was also an excess, of a solution of ammonio-acetate of zinc was then added, and the bulk of the mixture brought up to a given point by the addition of sufficient water; the precipitate formed was allowed to separate as far as possible by standing, and the mixture then boiled down at a low heat in a partial vacuum to half (or to any specified degree) of its original volume. After allowing it to cool thoroughly the

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\* Dingler's Polyt. Journal 227, p. 481.

precipitate was filtered out and washed as completely as possible with a known volume of water heated to about 80° C.; it was then dissolved in warm diluted sulphuric acid, titrated with the permanganate of potassium in presence of indigo-carmin,

No.	Hemlock.	Oak.	Chestnut.	Nut Galls.	Sumach.	
1	9.30	10.00	5.50	67.00	10.00	Catechu,
2	12.00	12.50	6.50	69.00	13.50	Kino
3	10.00	9.50	5.20	65.50	11.00	and
4	11.00	11.20	7.00	68.20	13.00	Rhatany,
5	8.50	9.00	6.00	60.00	9.00	
6	18.60	13.00	9.00	69.00	19.00	non-
7	11.00	12.00	6.00	66.00	11.00	accordant
8	20.30	15.00	11.00	72.00	20.00	results.
9	8.50	7.50	6.20	60.00	14.50	
10	3.00	5.20	2.80	47.50	3.50	
Average.	12.20	10.40	6.50	62.40	12.50	
Real value.	7.90	8.22	7.42	61.50	16.25	

and the result calculated from the data so obtained as in Lowenthal's method. Above is a table of the results obtained, in percentages. The great differences which it will be noticed, occur so abruptly are principally caused by varying the conditions, and I will detail them individually. Result number one was obtained by having the amount of zinc solution, in excess, a minimum, and conducting the evaporation in the shortest possible time. Number two was obtained by omitting the preliminary ammonia treatment preceding the addition of the ammonio-zinc solution, and in exact accordance with the detail of the method as originally proposed and published in *Bolley's Handbuch*. It is the treatment with the ammonia that causes the great difference in these two results, as a fair amount of precipitate was in that way separated which would otherwise have been precipitated with the tannate of zinc by the ammonia then added. Result number three was obtained by titrating the solution before and after the separation of the tannic acid by the zinc under the same condition as in number one, thus the method was modified to the principle involved in Löwenthal's. Result number four was obtained in the same manner, except with the omission of the ammonia treatment. Notice the difference in the result. Returning now to the original mode, in number five the evaporation of the mixture was to only two-thirds of its volume, and in number six to one-third; numbers seven and eight were

respectively conducted under the same conditions as numbers five and six omitting the preliminary ammonia treatment. Two blank samples of decoction were taken in this connection and evaporated down to one-third of their original volume, one after the preliminary treatment with ammonia the other omitting it. In both cases considerable matter deposited, although more in the latter than in the former, thus showing how numbers six and eight have been influenced. Result number nine was obtained by using almost the exact amount of zinc solution necessary, evaporating at a very moderate rate down to two-thirds, and with the preliminary ammonia treatment. Number ten was obtained by just the reverse of number nine, with the exception that the evaporation, although very rapid, was down to the same point. All the figures together have a value of 85% ; result number nine a value of about 82%, higher by far than any single one of the other results, but still too low for accuracy, as a confirmatory duplicate of this result yielded a value differing by 10% at least. The causes of the discrepancies are these: The compound formed with the zinc is quite soluble in an excess of zinc solution; this was ascertained by several trials. Besides this error, which in number ten is shown to be considerable, in the concentration of the mixture other matter precipitates mixed with the zinc compound besides that which would be thrown down by the preliminary ammonia treatment, although I have found that the matter which has the tendency to precipitate when the tannic acid, its solvent, is abstracted from the solution, is held in solution by the free ammonia present, and thus in this and my own method that source of error is not introduced to the slightest extent, while, as I showed before, it so markedly influences the results of Hammel's and Löwenthal's method. In boiling the mixture down at too high a heat the ammonia is evolved and this matter besides the gallate of zinc, etc. precipitates for want of the solvent. This is well shown by the lower result of number nine, which, as I before noticed, was evaporated more slowly than number one.

Take this method and modify it by the preliminary treatment with ammonia and filtration, which is shown by the above reasoning to be of such importance, use little or no excess of ammonio-zinc solution, by finding the quantity necessary by a preliminary treatment, evaporate the mixture down by one-third of the original volume at a moderate rate, and thus results similar to number nine may be obtained. I will notice here that the tannate of zinc formed in this process, as well as the tannate of copper, as I have shown in a pre-



vions paper, are quite free from extraneous matter when from the oak, hemlock, galls, etc., but from the catechu, kino, etc., are very markedly contaminated, and thus the notice in the last column. As the examinations in this line were not continued, on ascertaining the first few results, with spent barks and liquors this method yielded too unsatisfactory results to be used at all and thus I am unable to tabulate my conclusions as I did of Læwenthal's and Hammel's methods, but I might add that it compares favorably with either in all its relations.

The next method examined is the gravimetric gelatin method, which is objectionable as not being a volumetric one. When first proposed by Sir Humphrey Davy this method was much used, and is in fact up to this day in more or less constant use in the primitive condition; but after Watts published his researches upon the numerous inaccuracies, showing that the tannate of gelatin was exceedingly soluble in an excess of its precipitant, taken together with the difficulty encountered in filtering it off, owing to its slimy nature, it lost favor, and was seldom used by scientists. Some time ago Stoddart showed that by adding alum to the solution, both of these difficulties might be avoided to a great extent and fair results obtained, although if not of the absolute amount of tannic acid present, between this and the amount available for the formation of leather. But taken under any condition, this method, as I will show, cannot be compared with Hammel's, or much less Læwenthal's. In the examination of the method a series of five determinations each were made, and the results shown by the table below in percentages. All the causes of these grave errors, excepting the one I will notice below, are of the same character as those I detailed under Hammel's method, but without the introduction of extraneous matter, as when using hide. They have acted to a much more limited extent in this method, however, and thus the difference in the results.

No.	Hemlock.	Oak.	Chestnut.	Nut Galls.	Sumach.	
1.	5.30	6.20	3.50	50.20	10.00	Kino,
2.	4.20	3.80	3.60	52.00	13.00	Catechu and
3.	6.00	5.50	4.90	54.00	11.00	Rhatany, non-
4.	3.70	4.50	6.40	51.50	9.50	accordant re-
5.	4.50	4.30	5.80	56.00	14.00	sults.
Average,	4.70	4.80	4.70	52.90	11.50	
Real value,	7.90	8.22	7.42	61.50	16.25	

Owing to the influence of a cause comparatively absent in Hammel's method, the results are not solely affected by these inaccura-

cies. This cause is the solubility of tannate of gelatin, even in presence of the acids of Lœwenthal's method, though a little less there if alum has acted to such a degree as to make the results as low as some of the lower ones of Hammel's method.

Besides this, it may be well to recall to your attention here, that a solution of gallic acid is a powerful solvent, and especially in relation to this precipitate. The figures for the table were obtained in the following manner: The decoction was taken in measured quantity, mixed with sufficient alum solution and filtered from the resulting slight precipitate. A trial precipitation was first made to ascertain the approximate quantity of gelatin solution necessary, and then to another portion the necessary amount was added. The mixture, after a violent agitation, is set aside over night, and upon the next morning the precipitate is filtered off and washed with sufficient water at 5°C. After draining over the pump it is dried at 90°C. and weighed in the filter paper, the weight of which is known. The results in the table show this method to yield entirely too variable, and at the same time low figures, and therefore that the method cannot be employed in any determination where accuracy and speed are sought. The table shows a value of 87 %, I may add, but varies from this to 30 %. With "spent liquors" and "barks" the results are still more erroneous. Below are five determinations of the value of "spent liquors" in percentages of their weight:

No.	Hemlock.	Oak.	Sumach.	Catechu.
1	Hardly determinable.	0.10	.90	.50
2	" "	0.10	.40	.60
3	" "	0.10	.70	.50
4	" "	0.10	.50	.70
5	" "	0.10	.60	.60
Average,		0.10	.62	.60
Real value,	0.21	0.27	1.62	1.90

These figures do not afford me the necessary data to reason from in relation to the control, which is in this method thus unattainable.

The next method examined was the volumetric modification of the above. This method, which is even in use at this time, is nearly as inaccurate, and although a volumetric method, even as tedious as the other. The principal difficulty in the execution, and also in the accuracy of this method, is distinguishing the point at which there is no excess of either gelatin or tannic acid. This is literally impossible, as in the titration we encounter a condition in which

either the one or the other will produce a marked opalescence, and this lasts until a cubic centimetre of the one has been added in excess. This in itself condemns the method, besides there are the same inaccuracies which influence the results of the first and last method discussed. Another thing to be considered in both this and the preceding methods is, that tannic acid combines with a proportion of gelatin depending upon the amount of tannic acid present, or rather remaining in solution. To express it simply, the first portion of tannate of gelatin precipitating contains a much greater proportion of tannic acid than that falling when the quantity of the latter has decreased. Thus very variable amounts of the titrating solution may be used under varying degrees of dilution, and less or greater additions of it. In obtaining the results shown below in the table, the volumes of the standardizing solutions and those under examination were kept equal, as was also their approximate strength. The gelatin solution was added in c. c.'s at a time, and thus similar conditions existed as far as possible. The first five results were obtained from comparison with a standard solution of tannic acid, containing one gramme to a litre, and the others with the regular solution prescribed before for the other determinations. The mode of procedure was essentially as follows: The decoction in convenient quantity was placed in a beaker standing upon a blackened plate, and the gelatin solution added from the burette in c.c.'s at a time, at first at intervals of ten minutes, then of twenty minutes, and finally of one hour; when the end reaction was approximately found by taking out drops on a glass plate placed over some black glazed paper:

No.	Hemlock.	Oak.	Chestnut.	Sumach.	Nut Galls.	Catechu.
1	3.50	4.00	3.50	10.00	49.00	25.00
2	7.50	6.00	3.90	8.00	54.00	31.00
3	6.20	3.40	6.20	11.00	57.00	17.50
4	4.00	5.50	4.50	9.50	51.00	32.20
5	5.00	4.50	5.50	3.00	53.00	35.00
6	6.40	7.10	4.60	11.00	52.00	29.00
7	4.20	5.50	5.40	13.00	56.00	34.00
8	3.90	6.30	6.40	9.50	53.00	33.00
9	4.70	4.20	3.80	11.20	51.90	34.50
10	5.20	7.30	4.70	11.60	54.00	31.50
Average	5.10	5.43	4.85	10.78	53.20	30.27
Real value.	7.90	8.22	7.42	16.25	61.50	40.00

The average value thus deduced is 84 per cent., a little less than that of the others, but the figures are so discordant they condemn the method as little more than a mere surmise. In the determination of the strength of "spent barks" and "liquors" the results obtained are even of less value, as the following table will show: Numbers in percentage:

No.	Hemlock.	Oak.	Sumach.	Catechu.
1	trace	.15	.90	.80
2	.10	.20	.50	.90
3	trace	trace	.70	.80
4	.10	.10	.90	.70
5	.15	.15	.80	.90
Average,	.07	.12	.76	.82
Real value,	.21	.27	1.62	1.90

These figures were obtained as in the first five in the preceding table.

The next method examined was that of A. H. Allen, or rather Stoddart's modification of it. In the examination of this method the first five series of results were obtained by using swollen gelatine to separate the tannic acid, and in the last five by using a piece of hide properly cleansed, bated and raised. The general mode of procedure was as follows: A convenient amount of the decoction is measured out, and the standard solution of acetate of lead added, until a drop of the mixture filtered off ceases to color a mixture of ferricyanide of potassium and solution of ammonia with the characteristic red tint peculiar to the presence of, or in this case the excess of, tannic acid. Another portion of the decoction is then taken and the tannic acid separated from it as far as possible by the hide or swollen gelatin, as the case may be, and the remainder titrated as before. If the method was as accurate as might be supposed, no tannic acid would remain in the solution after this treatment, to afford the end reaction necessary in this titration, but as sufficient tannic acid does remain the end reaction is readily found. If the absorption of the tannic acid by the hide or gelatin was not so extremely tedious, and the errors noticed under Hammel's method more or less introduced, this method would rank far above Lœwenthal's, as the end reaction is perfectly perceptible when only  $\frac{1}{1000}$  part of tannic acid is present; and besides this, it is somewhat unsatisfactory to be necessitated to filter off small portions continually for observing the end

reaction. The results shown in the table below are very fair, but too high in the first five; and altogether unsatisfactory in the others.

Number.	Hemlock.	Oak.	Chestnut.	Nut Galls.	Sumach.	Catechu.	Kino.	Rhatany.
1	9.50	10.00	9.00	69.00	19.50	47.00	82.00	86.50
2	9.50	10.00	9.50	68.00	21.40	44.00	76.00	89.00
3	9.80	10.00	8.50	69.00	.....	.....	74.00	.....
4	9.60	11.00	10.00	67.00	.. ..	.. ..	.....	.....
5	9.40	10.50	9.00	67.50	.....	.....	.....	.....
6	7.10	6.50	11.00	52.00	14.80	51.00	64.00	42.00
7	11.60	9.80	6.00	67.00	21.00	38.00	.....	29.50
8	12.60	6.90	4.30	49.90	19.00	.....	.....	.....
9	6.40	8.40	9.20	57.00	.....	.....	.....	.....
10	9.20	11.10	7.00	67.50	.....	.....	.....	.....
Average,	9.37	9.37	8.35	63.30	19.10	45.00	74.00	87.00
Real value,	7.90	8.22	7.42	61.50	16.25	40.00	72.00	84.00

The value deduced from these figures is about 90 %. With "spent liquors" and "barks" the results obtained are even more fluctuating. Below is a table of a few determinations of "spent liquors";

No.	Hemlock.	Oak.	Sumach.	Catechu.
1	1.50	2.00	4.00	3.50
2	1.50	2.50	4.50	3.50
3	2.00	2.00	3.75	3.50
4	1.25	2.00	4.00	.....
5	2.00	2.25	4.00	.....
Average,	1.65	2.15	4.50	3.50
Real value,	0.21	0.27	1.62	1.90

in these the tannic acid was separated by gelatin, and these although more at variance with the truth than the others, are of some slight value when compared with Hammel's method. If in this method the tannic acid could be separated by some more appropriate process, it would yield even better results than any other now known. The next methods examined were those of Clarke and of Jean. The first is an ingenious method, using, as we know, a standard solution of cinchonia sulphate and magenta for an indicator, although yielding extremely poor results as at present executed. In fact, I saw almost immediately that nothing could be learned by examining it further than to justify these remarks. I believe that it is capable of favorable modification, and I will attempt to do this if possible. Of the last, Jean's, I can say but little; it, as some may know, consists of titrating with iodine, using starch as an indicator. The results obtained in a few preliminary

examinations were sufficient to discourage further investigation. It did not yield a value of over 60 per cent. Besides an error in the principle involved, an error is present similar to the principal one in Allen's method, that is, in the separation of the tannic acid by hide, etc., between the titrations of which six at least are necessary, even after the solutions are standardized (which also requires six), the first two for the approximate value, and the four others for the two final determinations.

To Mettenzway and Terriel we are indebted for the most ingenious as well as purely scientific method of determination, and one yielding far more accurate results than any which Proctor has examined. The method, I may add, consists in the deduction of the amount of tannic acid present by the amount of oxygen it is capable of utilizing to become oxidized. This method, however, has the insuperable disadvantage connected with it of having a very difficult mode of procedure, and is only successful under the careful manipulation of experienced hands, thus rendering it impossible except under special circumstances. Some time ago, but not in view of these investigations, I examined into its accuracy and formed these conclusions. In time I propose to modify this method if possible, to render it more simple in detail.

The next and last method examined was that of my own modification of Lœwenthal's and of Fleck's methods which I proposed some time ago before this society. I have given this method a most thorough and prolonged series of examinations, carefully controlling the results with working tests on a large scale. As it would take up too much space to detail the entire result of my investigations upon this method, I will only give a portion of them fairly and impartially selected, and at the same time from those executed under the the same conditions as were the preceding examinations. I will not detail my mode of procedure, as I have discussed it before the society at previous meetings, but I will here remark that the preliminary treatment of the decoction with diluted sulphuric acid is absolutely necessary, as the ellagic acid thus separated would otherwise interfere seriously with the copper precipitation, and would also call attention to the necessity of a preliminary ammonia treatment and filtration as in Carpené's method. In relation to the titration the suggestion of Proctor is available here. He suggests that the titration be conducted in a white porcelain dish and the first occurrence of the red or pink tint be noted, rather than

the yellow which is looked for when the titration is conducted in a flask. This red or pink tint is formed by the addition of one drop of the permanganate solution in excess and is observed around the edge of the apparently colorless space, where the depth of the solution is slight, next to the porcelain on the sides of the dish. I also wish to call attention to the necessity of using twice as much permanganate for the solution of indigo carmine alone, as is necessary for the tannic acid, etc. In the solution or decoction, as the case may be, I prefer to obtain my figure not by comparison with oxalic acid but under corresponding conditions with absolutely pure tannic acid.

No.	Hemlock.	Oak.	Chestnut.	Nut Galls.	Sumach.	Kino.	Catechu.	Rhatany.
1	7.80	8.10	7.50	59.50	17.00	86.00	45.00	57.00
2	7.85	8.10	7.50	59.00	16.50	84.00	45.00	55.50
3	7.75	8.15	7.50	58.50	16.90	86.00	45.00	58.00
4	7.95	8.25	7.45	59.00	16.75	85.00	45.00	57.00
5	8.05	8.20	7.50	59.00	16.40	86.00	45.00	59.00
6	7.85	8.10	7.40	59.00	15.95	84.00	44.50	56.00
7	7.80	8.30	7.35	60.00	16.20	85.00	46.00	56.50
8	7.90	8.25	7.45	59.00	17.00	85.00	45.00	55.00
9	7.75	8.10	7.40	60.50	16.50	84.00	45.00	54.00
10	7.70	8.20	7.50	59.50	16.50	86.00	46.00	56.50
Average, Control	7.83	8.15	7.45	59.20	16.57	85.20	45.15	56.70
or real value,	7.90	8.22	7.42	61.5	16.25	72.00	40.00	34.00

I will not make any remarks on this table except that it has a comparative value of 95% altogether, or 97% in the first five series. With "spent liquors" and "barks" the results are equally satisfactory.

No.	Hemlock.	Oak.	Sumach.	Catechu.	Hemlock.	Oak.
1	.20	.250	1.50	1.95	1.50	.90
2	.20	.260	1.50	2.00	1.40	.95
3	.19	.250	1.50	2.00	1.50	.90
4	.21	.280	1.55	1.90	1.50	.90
5	.20	.250	1.50	1.95	1.40	.90
Average,	.20	.255	1.51	1.96	1.46	.91
Real value by control,	.21	.270	1.62	1.90	1.50	.90

The first four are "spent liquors," the last two columns are of "spent bark."

Taking all these figures together and comparing the results with the working tests as under Löwenthal's and Hammel's methods,



it will be found that they are within 5% of the real value in all the figures, and within 3% in the first five columns, oak, hemlock, etc. From this it may be seen that the error in the execution of this method in ascertaining the percentage of tannic acid in an oak, hemlock, sumach, etc., would not exceed one  $\frac{1}{4}$  of one per cent., while in the estimation of catechu, kino, etc., the error is quite large. The cause of this is that the tannate of copper precipitated from these last materials is contaminated with considerable other matter, and besides this the peculiar principles present in these materials exert a solvent action upon the tannate of copper. This method may be used for these last determinations, but the kino may be more accurately determined by Lœwenthal's method.

This method has been in use in my laboratory ever since I first investigated it, and the numerous analyses I am called upon to make of tanning materials and executed by it give the utmost satisfaction. I will continue to use it in all my leather researches now about to be commenced, also in commercial work. In conclusion I wish to tender my thanks to Mr. Jackson S. Schultz for valuable advice, and to all others who favored me with materials for this research, and facilitated it.

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## REVIEW OF INDUSTRIAL CHEMISTRY.

BY A. A. BRENNEMAN.

SULPHURIC ACID.\*—*Lunge* has undertaken to study against the phenomena of the lead chamber in order to test the conclusions put forward by R. Weber (†) in 1867 as to the action of  $\text{SO}_2$  on  $\text{NO}$  and  $\text{N}_2\text{O}_5$ . He finds that  $\text{SO}_2$  and  $\text{NO}$  do not react upon one another, even at  $100^\circ \text{C}$ . when both are dry, but in presence of water they act slowly, the reaction requiring 48 hours for its completion at  $15^\circ$ .  $\text{N}_2\text{O}$  is formed, but the reduction does not extend further, no N being detected. Sulphuric acid of 1.45 sp-gr. does not hasten the reaction unless free O is also present.  $\text{N}_2\text{O}$  is produced in the latter case even when O is in excess of the amount required by the reaction  $2 \text{SO}_2 + 2 \text{NO} + 3 \text{O} + 3 \text{H}_2\text{O} = 2 \text{H}_2\text{SO}_4 + 2 \text{HNO}_2$ .

In the normal condition of the chamber both steam and O are in excess and a loss of nitrogen corresponding to the nitrate used in

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(\*) Ding. Jour. 243.1; Ber. D. Ch. Ges 14—2196. Chem. C. Bl. Dec. 7, '81.

(†) Ding. Jour. 184-246.



the pyrites burned must result, as  $\text{N}_2\text{O}$  is not absorbed by strong sulphuric acid in the Gay-Lussac tower.

The author also describes a new form of gas burette used in the investigation. *Laske* and *Benker* (\*) propose to reduce the waste of nitric acid in the chamber process by two-thirds through the introduction of  $\text{SO}_2$  at the outlet of the last chamber.  $\text{NO}_2$  being reduced by this means to  $\text{N}_2\text{O}$ , which is more soluble in  $\text{H}_2\text{SO}_4$ , complete absorption occurs in the Gay-Lussac tower.

*Richter* (†) effects thorough mixture of the gases of the lead chamber by aspirating them through one opening in the wall of the chamber and injecting them again at another. A steam jet is used for the purpose.

**SODA.**—*Scheurer-Kestner* (‡) discusses the method of *Pauli* (§) for the oxidation of soda lyes. The oxidation of sulphites to hypsulphites and sulphates through the action of  $\text{MnO}_2$  is considerable but still incomplete at a temperature of  $75^\circ$ .

*Rammelsberg* (||) finds in the soda liquors from the manufactory of Schoenebecker, peculiar red and yellow crystals containing vanadium and yielding, on re-crystallization, colorless octahedra of the general formula,  $\text{NaF} \cdot 2 \text{Na}_2 \text{RO}_4 \cdot 18 \text{H}_2\text{O}$ , in which R may be P or V. He ascribes the origin of the phosphorus to the limestone, the ash of coal and to the clay of the furnace, the latter being also the source of the vanadium.

The same author (¶) finds crystals of Gay-Lussite ( $\text{Na}_2\text{CO}_3 \cdot \text{CaCO}_3 \cdot 5 \text{H}_2\text{O}$ ) in soda liquors from the above works and ascribes to the formation of this substance one of the principal losses of soda in the Leblanc process.

*Reidemeister* (††) finds the same substance to separate from crude soda lyes during carbonation, but never from the fully carbonated liquor. It also occurs in the lime mud of the causticising vats in the manufacture of caustic soda. It is insoluble in water, but is decomposed by prolonged action of cold water, more readily with the aid of heat. It forms most abundantly in concentrated caustic liquors. An anhydrous soda-lime carbonate occurring in over-

(\*) Comptes Rendus, 92—181. Ding. Jour. 243.1.

(†) Ding. Jour. 243.1.

(‡) Ding Jour. 235-399.

(§) Comptes Rendus, 92.878. Ding. Jour. 242.4.

(||) Ding. Jour. 242.4.

(¶) " " "

(††) Ding. Jour., 242.—4.

heated or "burnt" balls in the black ash process gives crystalline structure to the mass and hinders subsequent lixiviation. The temperature of the black ash furnace should therefore be maintained at a point below the melting point of this double carbonate.

*W. Smith* and *T. Liddle* (\*) have shown that the loss of soda in the residue left from the lixiviation of black ash may be lessened, and the soda recovered by prolonged digestion with cold water, which decomposes the insoluble Gay-Lussite. "Soda waste" heated one hour at  $60^{\circ}$  yielded .22 per cent.,  $\text{Na}_2\text{O}$  out of 2.4 per cent. of soda present, and a sample of causticising mud yielded by the same treatment 2.62 per cent. of soda, while 2.5—3 per cent. remained insoluble. By using this mud to replace lime in the balling process the insoluble soda is recovered, but when the mud is rejected a loss as high as 7 per cent. of soda may occur. Wright has already shown that the total loss of  $\text{Na}_2\text{O}$  in the Leblanc process may amount to 20 per cent. The writers confirm the statement of Scheurer-Kestner that an excess of lime in the balling furnace favors the production of insoluble compounds of soda during lixiviation.

The manufacture of soda by the ammonia process of Solvay has been improved by *Honigman*(†), who employs a moderately elevated temperature in the absorbing vessel, securing by this means a granular precipitate of bicarbonate instead of the slimy mud commonly produced in the cold. The precipitate is easily washed. After washing, it is pressed in the cakes which are decarbonated in an upright furnace, on the grate of which the cakes are exposed to a current of hot  $\text{CO}_2$  from the generator. The dry powder falls through the grate, and the additional volume of  $\text{CO}_2$  obtained is carried to the absorber.

Improvements in the apparatus of the ammonia process have also been made by *Montblanc and Gaulard* (‡), *Pechiney* (§) and by the *Societe Anonyme de Produits Chimique de Paris* (||). *Ortlieb* and *Muller* (¶) apply the method of Solvay to the production of potassium carbonate, also through the substitution of trimethylamine for ammonia.

Trimethylamine, through the recent discovery of Camille Vincent, has become an abundant commercial product, being made

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\* Chem. News, No. 43. Ding. Jour., 242.4.

(†) Ding. Jour., 243.-1.

(‡) Ding. Jour. 243.1. (§) Ib. (||) Ib.

(¶) Bul. Soc. Chimique, Jan. 5th, '82.

from the residues of beet root molasses. A single firm, Tilloy & Dulanne, of Coumeres, turn out 1800 kilos of crude salts of trimethylamine per day.

A current of  $\text{CO}_2$  is passed into a solution of equal equivalents of potassium chloride and neutral trimethylamine hydrochloride at  $23^\circ \text{C}$ . and potassium carbonate ( $\text{KHCO}_3$ ) is precipitated. The reaction is limited by the inverse reaction, but this difficulty is overcome by operating under pressure. An excess of trimethylamine (2.22 molecules to 2  $\text{KCl}$ ) has been found to increase the yield.

*Lunge* (\*) has directed the examination of several reactions which have been suggested as indicating new methods of making soda, or of improving existing methods. The results are given as follows:

1. The action of  $\text{Al}_2\text{O}_3$  on  $\text{NaNO}_3$  at high temperatures with recovery of the nitric acid causes rapid attack of the vessels (platinum, iron and glass used) and  $\text{HNO}_3$  is only partly recovered. The reaction is less available for manufacturing purposes than the earlier one of Schiappi, in which  $\text{CaCO}_3$  and  $\text{NaNO}_3$  are used.

2. The decomposition of sodium sulphate by a solution of calcium bicarbonate (*Pongowski's method*) (†) was found impossible using a three-fold quantity of pulverized marble, acting for ten hours upon sodium sulphate in a solution saturated with  $\text{CO}_2$ .

3. Aarland's process for substituting  $\text{CaCl}_2$  for  $\text{MgCl}_2$  in treatment of soda waste by the well known method of Schaffner and Helbig failed entirely when tried in the laboratory.  $\text{CaCl}_2$  decomposes  $\text{Ca S}$  only after prolonged boiling, a result which may be obtained with water alone.

POTASH.—*Gruneberg* (‡) reviews the progress of the Stassfurt industry in its relation to the production of potassium sulphate from the complex salts and mixtures of the natural deposit. He discusses a list of 20 patents which have appeared since 1877, and classifies them as follows:—

1. Preparation of Schoenite ( $\text{MgSO}_4, \text{K}_2\text{SO}_4, 6\text{H}_2\text{O}$ ) from Kieserite ( $\text{MgSO}_4$ ) and  $\text{KCl}$  or Carnallite ( $\text{K Mg Cl}_3, 6\text{H}_2\text{O}$ )

2. Preparation of Schoenite from Kainite.

3. Working of mother liquors for by-products.

4. Production of  $\text{K}_2\text{SO}_4$  from Schoenite by methods other than those depending on decomposition of  $\text{KCl}$ .

The author concludes that a combination of different processes

(\*) Ding. Jour. 243 Heft. 2.

(†) French Patent, Mar. 27, 1872.

(‡) Ding. Jour. 243 Heft. 1.

will be found best in practice. In all processes it is advisable to use a minimum of water, and to have all waste liquors as rich in  $\text{MgCl}_2$ , and as poor in potassium salts as possible before they are rejected.

All methods of preparing potassium sulphate from these salts must compete with the direct method of the Leblanc process, by which the alkaline chloride is converted into sulphate, and are at an additional disadvantage in comparison with it, because of the loss of  $\text{HCl}$  in the waste liquors.

## ABSTRACTS.

Abstracts from the *Comptes Rendus*, by A. Bourgougnon.

**Complex Function of Morphine, and its Transformation into Picric Acid; also its Solubility.** BY M. CHASTAING. No. 1. (Jan. 2nd, 1882).—Action of alkalies. Potassa, soda, baryta and lime dissolve morphia in the proportion of one equivalent of morphia for one equivalent of base. The solutions evaporated in a vacuum give crystallized products containing one equivalent of base, one equivalent of morphia and two equivalents of water.

Action of Nitric acid. Tetrahydrated nitric acid at a temperature of  $100^\circ \text{C}$ . transforms morphia into an acid with the formula  $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_{18}$ , a tetrabasic acid crystallizing with difficulty. Baryta and lead salts of this acid contained  $8\text{H}_2\text{O}$ .

Monohydrated nitric acid, in sealed tubes at  $100^\circ \text{C}$ . transform the acid  $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_{18}$  into picric acid.

Solubility of morphia. 1 litre of water at  $0^\circ \text{C}$ . dissolve traces of morphia, at  $10^\circ$  0 gr. 10; at  $20^\circ$  0 gr. 20; at  $40^\circ$  0 gr. 40, of crystallized morphia. The solubility of morphia at  $40^\circ$  is then represented by a straight line, but above  $45^\circ$  the solubility is represented by a larger number than the elevation of temperature. From  $45^\circ$  to  $100^\circ$  the curve of solubility affects a parabolic form.

**Carbonic Ether of Borneol.** BY M. A. HALLER. No. 2. Jan. 9th, 1882.—This ether was found in the residues of preparation of the compound described by the author under the name of *Borneol Cyane*. When pure it affects the form of very light white needles or hexagonal tables, insoluble in water and the alkalies, not very soluble in cold alcohol, soluble in boiling alcohol, ether, chloroform, benzine, glacial acetic acid. Melting point  $215^\circ \text{C}$ . and sublimes without decomposition. Melted with caustic potash it gives potassium carbonate and borneol.

In the cold, nitric acid is without action, at  $100^{\circ}$  C. a combination takes place with the formation of an oily substance, if the temperature is raised above  $100^{\circ}$  there is a decomposition with formation of nitrous vapors and production of camphor. Formula,  $C_{21}H_{34}O_3$ .

**Essential Oil of Savory.** BY M. A. HALLER. No. 3. (Jan. 16, 1882.)—This essential oil is obtained from Savory, (*Satureia Montana* L), it is a mixture of hydrocarbons and phenols, and does not contain camphor; it is a liquid of an orange color, not very fluid, of an aromatic odor. Sp. gr. 0.7394 at  $17^{\circ}$  C. Rotatory power for a stratum of 200 <sup>m.m.</sup> is  $LD = -6^{\circ}5$  at  $17^{\circ}$  C. Treated with caustic soda an insoluble hydrocarbon is produced; if after the separation of the insoluble hydrocarbon, the alkaline solution is acidified, an oily liquid is produced which distils between  $230^{\circ}$  and  $245^{\circ}$  C. After several rectifications a liquid is obtained boiling at  $232^{\circ}$ — $233^{\circ}$  C. Sp. gr. 0.972 at  $17^{\circ}$ , soluble in alcohol, ether and alkalies. It has for formula  $C_{10}H_{14}O$  and is isomeric with thymol.

**Diatomic Alcohol Derived from Beta-Naphthol.** BY G. ROUSSEAU. No. 3. (Jan. 16, 1882.)—Beta-naphthol treated with chloroform and caustic soda in solution gives, besides an aldehyde, a white product characterized as a pseudo-diatomic alcohol.

It melts at  $230^{\circ}$  C. with decomposition, nearly insoluble in benzine, carbon disulphide and acetic acid, it is dissolved in small quantity in ether and petroleum naphtha.

An analysis has given the following numbers.

	I.	II.
C	86.01.....	85.94
H	4.65.....	4.60

The calculation for  $C_{22}H_{14}O_2$  gives

C	85.16
H	4.51

**Phosphoric Acid in the soils of the North of France.** BY A. LADUREAU. No. 3. (Jan. 16, 1882.) **Silicomolybdic Acid.** BY F. PARMENTIER. No. 5. (Jan. 30th, 1882.)—Obtained by the action of hydrochloric acid upon silicomolybdate of suboxide of mercury, the liberated acid gives by evaporation large bright-yellow cubo-octahedral crystals melting at about  $45^{\circ}$ , very soluble in water and diluted acids, forming crystallized salts with bases. Its composition is represented by  $SiO_2 \cdot 12 MoO_3 + 26 H_2O$ , corresponding to the silicotungstic acid of Marignac.

**Specific Gravity of Vapor of Pyrosulfuryl Chloride.** By J. OGIER. No. 5. (Jan. 30th, 1882.)—Victor Meyer's and Dumas' method have been employed.

The first method gives the following numbers: 3.88, 3.59, 3.99, 3.69, 3.36; mean 3.70 in anilin vapor. The results obtained in sulphur or mercury vapor do not differ very much; they are 3.72, 3.42, 3.30. By Dumas' method the numbers obtained are 3.87, 3.72, 3.69, 3.70; average 3.74 at ordinary pressure, and at temperatures between 160° and 200°C.

The pressure being reduced (200 to 300<sup>mm</sup> of mercury) in order to obtain a decrease of temperature, the sp. gr. obtained at 170° were 3.69, 3.75, 3.80, 3.69, average 3.73. The vapor weighed was ascertained by analysis to be a pure product.

**Researches on Pilocarpin.** By M. CHASTAING. No. 5. (Jan. 30th, 1882.)—Pure pilocarpin has been treated with melted potassa; a volatile product with an alkaline reaction was formed, giving a precipitate with a slightly acid solution of platinum chloride, forming methylamin chloroplatinate. There has been no formation of a volatile base having the characteristics of conicin. Under the influence of melted potassa, in excess, pilocarpin furnishes methylamin, carbonic acid, butyric acid, and traces of acetic acid.

## FOREIGN PATENTS.

C. ARNOLD, New York: *Apparatus for generating ozone* (Germ. P. 15678, Jan. 20, 1881). Into a tray of glazed earthenware are placed vessels containing pieces of phosphorus and water. The tray is covered with a bell of unglazed, porous earthenware. Through this bell the air passes in and the ozone passes out whilst the oxidation products of the phosphorus remain inside and dissolve in the water contained in the glazed tray.

H. PRECHT, New Stassfurt: *Method of preparing magnesia, potassium sulphide and potassium sulphate from potassium-magnesium sulphate*. (Germ. P. 15,747, March 8, 1881). The manufacture of potassium sulphate from the above mentioned double sulphate by heating it to redness by carbon, according to the equation:—  

$$2 (\text{K}_2\text{SO}_4 \text{ MgSO}_4) + \text{C} = 2 \text{K}_2\text{SO}_4 + 2 \text{MgO} + 2 \text{SO}_2 + \text{CO}_2;$$
as indicated by Schwarz (Dingler's polyt. J. vol. 219, p. 352), is

modified so as to first produce potassium sulphide in the mixture. This sulphide effects the decomposition of the double sulphate with greater energy, rapidity and completeness.

I. EGESTORFF, Hanover: *Improvements in salt evaporating pans and method of filtering brine.* (Germ. P. 14,782, Feb. 1, 1881). Mechanical arrangement for protecting the steam coils from the depositing salt, and filtration of the hot brine through flannel before running it into the pan.

A. R. PECHINEY, Salindres: *Method of preparing chlorates.* (Germ. P. 15,493, March 10, 1881). By the usual method of manufacturing potassium chlorate, decomposition of the product resulting from the action of chlorine upon milk of lime, by means of potassium chloride, a part of the potassium chloride remains dissolved in the calcium chloride mother liquor, after the crystallization of the potassium salt. In order to reduce this loss to a minimum the inventor removes the greater part of the calcium chloride from the mixture of calcium chlorate and chloride before adding the potassium chloride. This he effects by evaporating the liquid to 48°Be. and cooling, whereupon calcium chloride crystallizes, and can be removed, or by adding 3 molecules of lime for 1 molecule of calcium chloride whereby basic chlorides are formed, which can be separated from the liquid, or finally both operations may be employed consecutively.

C. LUCKOW, Deutz: *Electrolysis of zinc solutions for the purpose of extracting zinc from its ores.* (Germ. P. 14,256, April 20, 1880). Concentrated solutions of zinc salts are electrolyzed. The zinc is separated in grains, and the acids liberated are made to act upon crude or wasted zinc ores connected with the anode of the battery. Polarization of the electrodes is neutralized by chemical or mechanical means.

A. GURLT, Bonn: *Method of working up calamine residues and zinc waste by treatment with gaseous or liquid hydrochloric acid in combination with chlorides of the alkalies or alkaline earths.* (Germ. P. 15,012, Dec. 5, 1880, 1st additional patent to No. 8,116). The modification of the original patent consists in the employment of gaseous or liquid hydrochloric acid during the distillation of the zinc chloride, whereby the extraction of the zinc is facilitated.

P. MANHES, Lyons: *Manufacture of refined copper from copper mattes in Bessemer converters by the use of reducing agents.*



(Germ. P. 15,562, Aug. 20, 1880). Sulphurous copper ores are melted down in a pit furnace. The matte obtained is treated with air in a converter in the same manner as pig iron in the Bessemer process. The operation is ended when evolution of sulphurous acid has nearly ceased. The product is crude copper. To refine it, charcoal powder is blown in with the air. Modifications of the process are described for the treatment of mattes rich in iron or too deficient in sulphur to produce the required degree of fluidity.

MAX TSCHIRNER, San Francisco: *Improvements in the manufacture of explosive compounds*. (Germ. P. 15,508, Feb. 6, 1880). Picric acid, 1 part; tar, 1 part; and potassium chlorate, 5 parts.

L. M. LEWIN, Paris: *Use of cotton and dextrine for the preparation of a gelatinous nitro-glycerine*. (Germ. P. 15,073, Jan. 18, 1881). Purified and finely divided cotton is mixed with 5 parts of dextrine and heated under pressure in a closed vessel. The jelly thereby formed is dissolved in nitro-glycerine to the extent of 7 per cent., and forms a mass from which the nitro-glycerine will not drain. An explosive compound is formed by mixing this gelatinous nitro-glycerine with nitre and sawdust.

CHEMISCHE FABRIK AUF ACTIEN, Berlin: *Preparation of coloring matters from sulphosalicylic acid*. (Germ. P. 15,117, Dec. 10, 1880, and Germ. P. 15,889, Mar. 18, 1881). Sulphosalicylic acid is nitrated by means of nitric acid of sp. gr. 1.35. Stronger nitric acid eliminates the sulpho group with formation of various nitro-compounds. Nitro-sulphosalicylic acid and its soluble salts dye silk and wool yellow. The coloring power is augmented by the introduction of one atom of bromine. Excess of bromine also eliminates the sulpho group, forming dibromnitrosalicylic acid.

Sulphosalicylic acid also reacts with phenols to form coloring matters, yielding, for instance, with resorcin a brownish-red dye.

With diazo, diazoamido, and amidoazo compounds, sulphosalicylic acid produces red dyes.

In Patent No. 15,889, the method of preparation of the bromine substitution compounds of nitrosulphosalicylic and nitrosalicylic acids is claimed.



## UPON THE PREPARATION OF ARTIFICIAL INDIGO AND SOME OF ITS DERIVATIVES.

BY JAMES H. STEBBINS, JR., S. B.\*

It will be needless for me to tell you what indigo is, as you have probably all come across it, and know whence it is derived.

I will therefore only give you a graphic description of how this interesting body can be made synthetically; and in giving you this description, I can do no better than quote Baeyer's wonderful experiments :

Baeyer says : "The first thing I attempted to do was to remove the oxygen in isatine, and it will be remembered how this was accomplished, in 1865 and 1866, by the conversion of isatine into dioxindole, oxindole, and indole."

It was then tried to find an easier method for the production of indole, as Baeyer considered it to be the basis of the indigo group.

In 1868 Baeyer obtained it directly, by treating indigo with zinc dust ; and in 1869 he produced it synthetically from nitrocinnamic acid and its reduction product.

The following quotation may help to explain Baeyer's reasoning more thoroughly :

"If it is desired to make indole synthetically, it becomes necessary to introduce into benzol a diatomic carbon chain and a nitrogen atom, and then to connect the one with the other. The properties hereby necessitated will be found realized in nitrocinnamic acid, which has been deprived of carbonic acid, and the oxygen of the nitro group ; and, in fact, as we have already found, nitrocinnamic acid on being fused with potash produces indole."

Shortly after this, Kekule informed us that he had produced in a similar way isatine, from orthonitrophenylacetic acid. Baeyer and Suida then examined this compound more closely, and came to the conclusion that dioxindole and oxindole could be nothing else than the anhydrides of orthoamido-amygdalic acid and orthoamido-phenyl acetic acid. A synthetical examination soon after followed, and showed that oxindole by oxidation is converted into isatine ; which, however, was shortly afterwards produced by Claisen and Shadwell, by a new and convenient synthetical method.

In 1870, Baeyer and Emmerlin converted this isatine into indigo, and thus solved the problem he had so long been working at ; but, as the materials used (phosphorous trichloride, phosphorous, and acetyl chloride) were quite complicated, he strove to find a more simple method for accomplishing this end, and finally came to the

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\*Read at the January Conversazione.

conclusion that it would be better to first treat the isatine with phosphorous pentachloride, and then phosphorus, or some other reducing agent.

It was then tried to produce, in the same way, indole from oxindole; and, in fact, a body answering to the formula of a dichlorindole was obtained.

Without taking up any more of your time with the history of Baeyer's discoveries, I will now describe some of the products which may be converted into indigo blue.

#### ORTHONITROCINNAMAC ACID.

This acid may be easily prepared, according to Beilstein and Kullberg's process, and likewise the separation of the ortho from the paranitro compound by means of ether.

This acid bears a simple relation to indigo, as may be seen from the following formulæ :

*Nitrocinnamic Acid. Indigo.*



#### ORTHONITRODIBROMCINNAMIC ACID.

This acid is produced by treating the nitro compound with bromine under exclusion of sunlight.

The bromine compound crystallizes in short colorless needles, which melt at 180° C., under decomposition and formation of a small amount of indigo.

This body was found to have the formula  $C_9 H_7 (Br_2) NO_4 =$  orthonitrodibromcinnamic acid.

This body is soluble in caustic alkalies, without decomposition; which, however, by heat is converted into orthonitropropionic acid, and then into isatine. By heating an aqueous solution of the above acid with carbonate of soda, a small amount of crystallized indigo blue is the result.

#### ORTHONITROPHENYLPROPIOLIC ACID.



The bearing of orthonitrodibromcinnamic acid with alkalies differs considerably from the non-nitrated compound, inasmuch as HBr may readily be removed from the former, whereas from the latter it can only be removed by the energetic action of alcoholic potash to form propiolic acid. The propiolic acid is prepared usually from an aqueous solution of the dibrominated compound by treating with an excess of caustic soda solution.

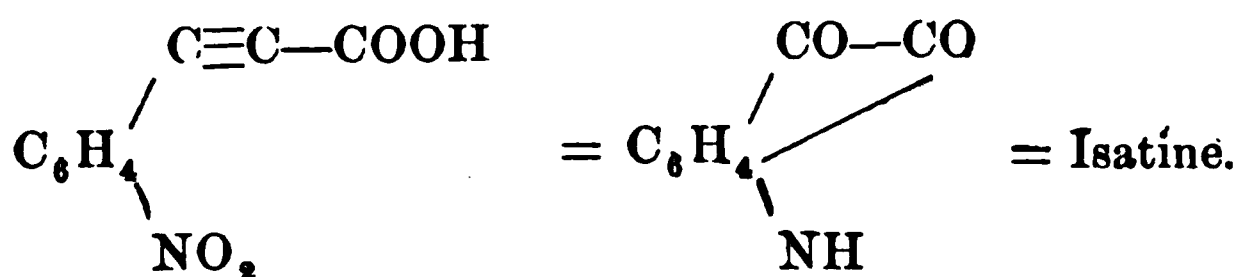
The compound is then thrown down by means of an acid, and dried. The orthonitrophenylpropionic acid so obtained appears as little colorless needles, melting at 155—160° C. with sudden decomposition.

#### ORTHONITROPHENYLACETYLEN.

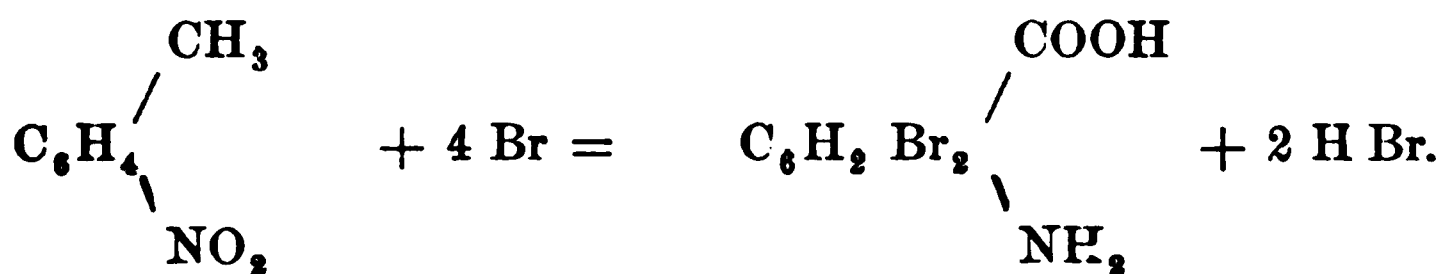
This compound may be obtained from orthonitrophenylpropionic acid, by boiling with water in an ascending cooler. It crystallizes from alcohol, in colorless needles, and melts at 81–82° C.

#### FORMATION OF ISATINE FROM ORTHONITROPHENYLPROPIOLIC ACID.

Isatine may be obtained in abundance by boiling a solution of orthonitrophenylpropionic acid with alkalis. The reaction that takes place here may be explained as follows :



In other words,  $\text{CO}_2$  is split off; at the same time a reduction of the  $\text{NO}_2$  group, and oxidation of the side chain takes place. This reaction is similar to the reaction which takes place when bromine is allowed to act on orthonitrotoluol, viz :

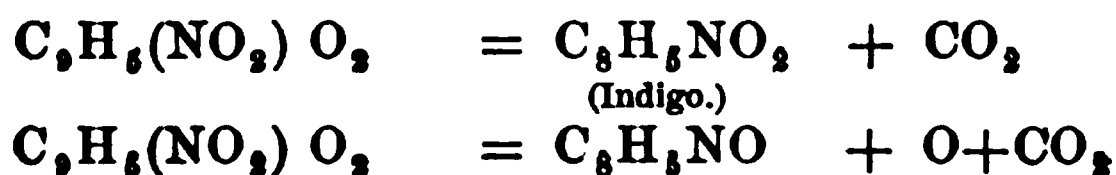


#### FORMATION OF INDIGO FROM ORTHONITROPHENYLPROPIOLIC ACID.

If a very dilute alkaline solution of the acid be heated to boiling, and a small piece of grape sugar is added thereto, the liquid first turns blue, and then follows a copious precipitation of small blue needles, with coppery red lustre. These needles are perfectly pure indigo-blue. The yield so obtained is about 40% of the propiolic acid used.

The formation of indigo-blue in this case takes place as follows :

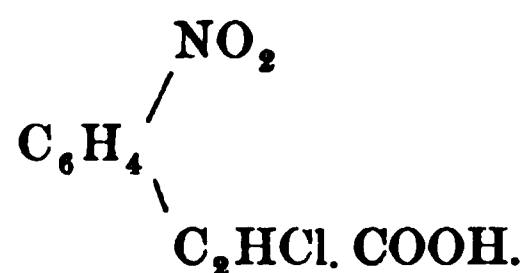
First the alkali, acting on propiolic acid, splits off  $\text{CO}_2$ , forming isatine, which, however, on being treated with a reducing agent as grape sugar, loses an atom of oxygen and forms indigo-blue, viz :



## ORTHONITROPHENYLCHLORLACTIC ACID.

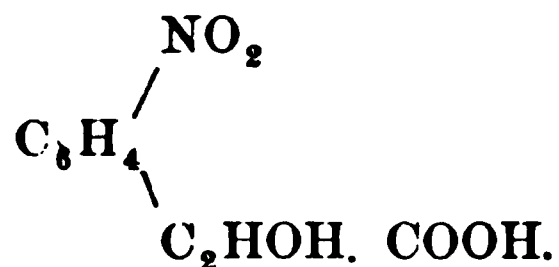
This acid may be easily prepared by passing a current of chlorine into an alkaline solution of orthonitrocinnamic acid. The mixture so produced is composed of orthonitrochlorstyrol, and orthonitrophenylchlorlactic acid, from which the latter may be separated by acidifying the mixture and then extracting the orthonitrophenylchlorlactic acid with ether. This compound appears as a white crystalline mass, and having a melting point of  $119^{\circ}$ — $120^{\circ}$  C.

Its formula may be represented by



This compound on being treated with reducing agents, such as sodium amalgam, or sulphate of iron in an alkaline solution, forms indole.

## ORTHONITROPHENYLOXACRYLIC ACID.



This compound may easily be produced from orthonitrophenylchlorlactic acid, by treating with alcoholic potash. It crystallizes from hot water, in flat needles.

If heated slowly to  $110^{\circ}$  C. it melts, giving off  $\text{CO}_2$ , and forming a dark blue mass, which, on being treated with alcohol, dissolves with a brown color, in which small crystals of indigo-blue remain suspended.

Indigo-blue may also be derived from the above by heating its solution with phenol, but the yield of coloring matter so obtained is quite small.

The reaction taking place here may best be explained as follows:

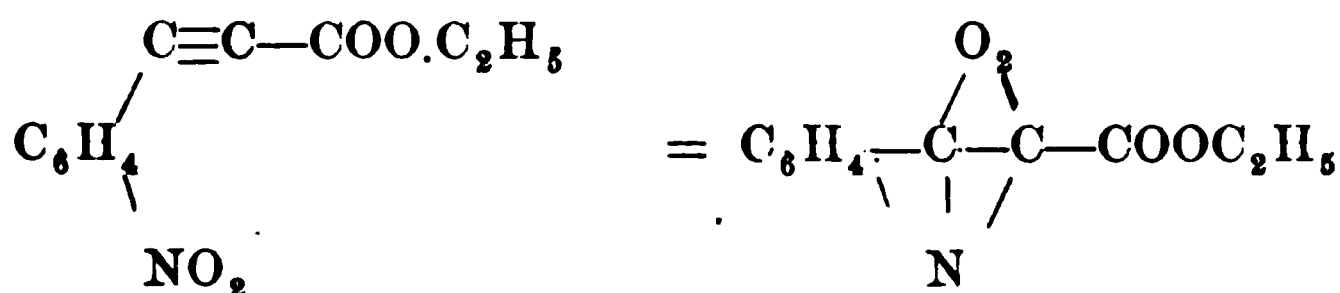


The ease with which orthonitrophenylpropionic acid is converted into indigo-blue, with such mild re-agents as grape sugar, appeared very strange to Baeyer, and in the following I will detail the experiments he made to arrive at a clear understanding of the above mentioned re-action.

By treating orthonitrophenylpropionic acid with strong  $\text{H}_2\text{SO}_4$ , at

the ordinary temperature, its isomer isatogenic acid ether is obtained, which crystallizes in yellow needles and melts at  $115^{\circ}$  C.

The formation of this compound may be explained by the following re-action :



A solution of nitrophenylpropionic acid in  $\text{H}_2\text{SO}_4$ , on being treated with reducing agents, forms a new blue dye-stuff, called indoine. Indoine is very similar to indigo blue but differs from it in that it dissolves in strong  $\text{H}_2\text{SO}_4$  with a blue color, which, on being heated, only forms a sulpho acid with great difficulty.

#### INDOXYLIC ACID ETHER.

This body is produced by the action of reducing agents, on isatogenic acid ether. It appears in the shape of thick prisms, and melts at  $120-121^{\circ}\text{C}$ .

It has the formula  $\text{C}_{10}\text{H}_{11}\text{NO}_3$ .

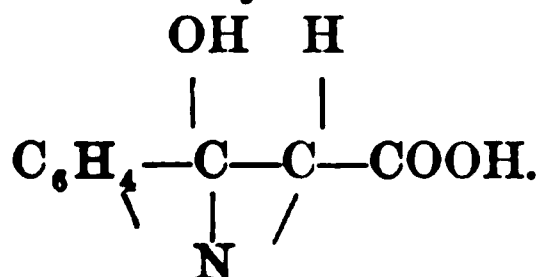
This compound on being rapidly heated forms a little indigo-blue; but if heated to  $100^{\circ}\text{C}$ . with strong  $\text{H}_2\text{SO}_4$  it passes over into indigo sulpho acid almost quantitatively.

On being fused with caustic soda, at a temperature of  $180^{\circ}$ , it forms indoxylic acid, which melts at  $160^{\circ}\text{C}$ .

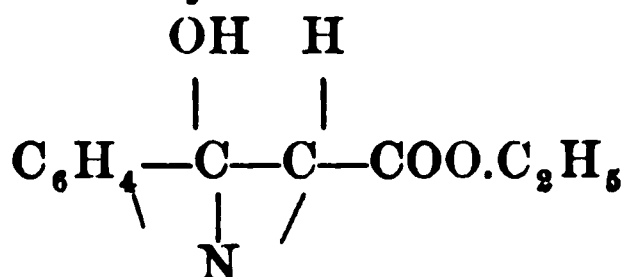
Ethyl indoxylic acid and nitrosoethylindoxylic acid have likewise been produced, but, I will not attempt to describe their formation.

The following formulæ will best represent the compounds just enumerated.

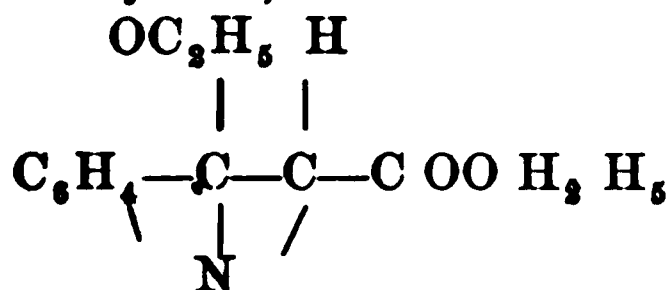
#### Indoxylic acid.



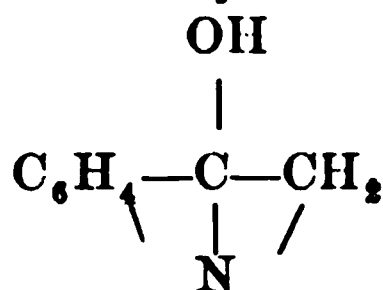
#### Indoxylic acid ether.



#### Ethylindoxylic acid ether.



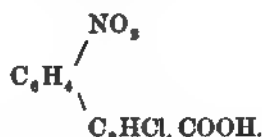
#### Indoxyl.



## ORTHONITROPHENYLCHLORLACTIC ACID.

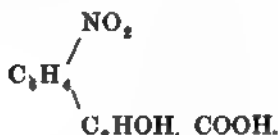
This acid may be easily prepared by passing a current of chlorine into an alkaline solution of orthonitrocinnamic acid. The mixture so produced is composed of orthonitrochlorstyrol, and orthonitrophenylchlorlactic acid, from which the latter may be separated by acidifying the mixture and then extracting the orthonitrophenylchlorlactic acid with ether. This compound appears as a white crystalline mass, and having a melting point of  $119^{\circ}$ — $120^{\circ}$  C.

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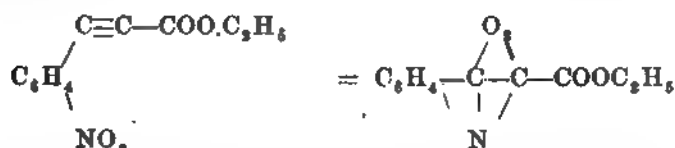


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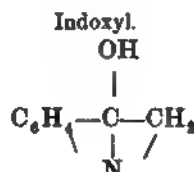
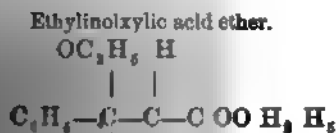
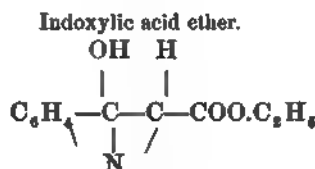
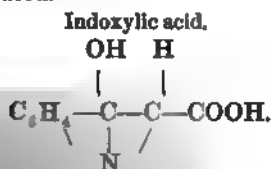
It has the formula  $\text{C}_{10}\text{H}_{11}\text{NO}_2$ .

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Ethyl indoxylic acid and nitrosoethylindoxylic acid have likewise been produced, but, I will not attempt to describe their formation.

The following formulæ will best represent the compounds just enumerated.



will be found best in practice. In all processes it is advisable to use a minimum of water, and to have all waste liquors as rich in  $\text{MgCl}_2$ , and as poor in potassium salts as possible before they are rejected.

All methods of preparing potassium sulphate from these salts must compete with the direct method of the Leblanc process, by which the alkaline chloride is converted into sulphate, and are at an additional disadvantage in comparison with it, because of the loss of  $\text{HCl}$  in the waste liquors.

## ABSTRACTS.

Abstracts from the *Comptes Rendus*, by A. Bourgougnon.

**Complex Function of Morphine, and its Transformation into Picric Acid; also its Solubility.** BY M. CHASTAING. No. 1. (Jan. 2nd, 1882).—Action of alkalies. Potassa, soda, baryta and lime dissolve morphia in the proportion of one equivalent of morphia for one equivalent of base. The solutions evaporated in a vacuum give crystallized products containing one equivalent of base, one equivalent of morphia and two equivalents of water.

Action of Nitric acid. Tetrahydrated nitric acid at a temperature of  $100^\circ \text{C}$ . transforms morphia into an acid with the formula  $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_{18}$ , a tetrabasic acid crystallizing with difficulty. Baryta and lead salts of this acid contained  $8 \text{H}_2\text{O}$ .

Monohydrated nitric acid, in sealed tubes at  $100^\circ \text{C}$ . transform the acid  $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_{18}$  into picric acid.

Solubility of morphia. 1 litre of water at  $0^\circ \text{C}$ . dissolve traces of morphia, at  $10^\circ$ , 0 gr. 10; at  $20^\circ$  0 gr. 20; at  $40^\circ$  0 gr. 40, of crystallized morphia. The solubility of morphia at  $40^\circ$  is then represented by a straight line, but above  $45^\circ$  the solubility is represented by a larger number than the elevation of temperature. From  $45^\circ$  to  $100^\circ$  the curve of solubility affects a parabolic form.

**Carbonic Ether of Borneol.** BY M. A. HALLER. No. 2. Jan. 9th, 1882.—This ether was found in the residues of preparation of the compound described by the author under the name of *Borneol Cyane*. When pure it affects the form of very light white needles or hexagonal tables, insoluble in water and the alkalies, not very soluble in cold alcohol, soluble in boiling alcohol, ether, chloroform, benzine, glacial acetic acid. Melting point  $215^\circ \text{C}$ . and sublimes without decomposition. Melted with caustic potash it gives potassium carbonate and borneol.



In the cold, nitric acid is without action, at 100° C. a combination takes place with the formation of an oily substance, if the temperature is raised above 100° there is a decomposition with formation of nitrous vapors and production of camphor. Formula,  $C_{21}H_{34}O_3$ .

**Essential Oil of Savory.** By M. A. HALLER. No. 3. (Jan. 16, 1882.)—This essential oil is obtained from Savory, (*Satureia Montana* L), it is a mixture of hydrocarbons and phenols, and does not contain camphor; it is a liquid of an orange color, not very fluid, of an aromatic odor. Sp. gr. 0.7394 at 17° C. Rotatory power for a stratum of 200 <sup>m.m.</sup> is  $LD = -6^{\circ}5$  at 17° C. Treated with caustic soda an insoluble hydrocarbon is produced; if after the separation of the insoluble hydrocarbon, the alkaline solution is acidified, an oily liquid is produced which distils between 230° and 245° C. After several rectifications a liquid is obtained boiling at 232°—233° C. Sp. gr. 0.972 at 17°, soluble in alcohol, ether and alkalies. It has for formula  $C_{10}H_{14}O$  and is isomeric with thymol.

**Diatomic Alcohol Derived from Beta-Naphthol.** By G. ROUSSEAU. No. 3. (Jan. 16, 1882.)—Beta-naphthol treated with chloroform and caustic soda in solution gives, besides an aldehyde, a white product characterized as a pseudo-diatomic alcohol.

It melts at 230°C. with decomposition, nearly insoluble in benzine, carbon disulphide and acetic acid, it is dissolved in small quantity in ether and petroleum naphtha.

An analysis has given the following numbers.

	I.	II.
C	86.01.....	85.94
H	4.65.....	4.60

The calculation for  $C_{22}H_{14}O_2$  gives

C	85.16
H	4.51

**Phosphoric Acid in the soils of the North of France.** By A. LADUREAU. No. 3. (Jan. 16, 1882.) **Silicomolybdic Acid.** By F. PARMENTIER. No. 5. (Jan. 30th, 1882.)—Obtained by the action of hydrochloric acid upon silicomolybdate of suboxide of mercury, the liberated acid gives by evaporation large bright-yellow cubo-octahedral crystals melting at about 45°, very soluble in water and diluted acids, forming crystallized salts with bases. Its composition is represented by  $SiO_2 \cdot 12 MoO_3 + 26 H_2O$ , corresponding to the silicotungstic acid of Marignac.

**Specific Gravity of Vapor of Pyrosulfuryl Chloride.** By J. OGIER. No. 5. (Jan. 30th, 1882.)—Victor Meyer's and Dumas' method have been employed.

The first method gives the following numbers: 3.88, 3.59, 3.99, 3.69, 3.36; mean 3.70 in anilin vapor. The results obtained in sulphur or mercury vapor do not differ very much; they are 3.72, 3.42, 3.30. By Dumas' method the numbers obtained are 3.87, 3.72, 3.69, 3.70; average 3.74 at ordinary pressure, and at temperatures between 160° and 200°C.

The pressure being reduced (200 to 300<sup>mm</sup> of mercury) in order to obtain a decrease of temperature, the sp. gr. obtained at 170° were 3.69, 3.75, 3.80, 3.69, average 3.73. The vapor weighed was ascertained by analysis to be a pure product.

**Researches on Pilocarpin.** By M. CHASTAING. No. 5. (Jan. 30th, 1882.)—Pure pilocarpin has been treated with melted potassa; a volatile product with an alkaline reaction was formed, giving a precipitate with a slightly acid solution of platinum chloride, forming methylamin chloroplatinate. There has been no formation of a volatile base having the characteristics of conicin. Under the influence of melted potassa, in excess, pilocarpin furnishes methylamin, carbonic acid, butyric acid, and traces of acetic acid.

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## FOREIGN PATENTS.

C. ARNOLD, New York: *Apparatus for generating ozone* (Germ. P. 15678, Jan. 20, 1881). Into a tray of glazed earthenware are placed vessels containing pieces of phosphorus and water. The tray is covered with a bell of unglazed, porous earthenware. Through this bell the air passes in and the ozone passes out whilst the oxidation products of the phosphorus remain inside and dissolve in the water contained in the glazed tray.

H. PRECHT, New Stassfurt: *Method of preparing magnesia, potassium sulphide and potassium sulphate from potassium-magnesium sulphate*. (Germ. P. 15,747, March 8, 1881). The manufacture of potassium sulphate from the above mentioned double sulphate by heating it to redness by carbon, according to the equation:—  

$$2 (K_2SO_4 \cdot MgSO_4) + C = 2 K_2SO_4 + 2 MgO + 2 SO_2 + CO_2;$$
as indicated by Schwarz (Dingler's polyt. J. vol. 219, p. 352), is

modified so as to first produce potassium sulphide in the mixture. This sulphide effects the decomposition of the double sulphate with greater energy, rapidity and completeness.

I. EGESTORFF, Hanover: *Improvements in salt evaporating pans and method of filtering brine.* (Germ. P. 14,782, Feb. 1, 1881). Mechanical arrangement for protecting the steam coils from the depositing salt, and filtration of the hot brine through flannel before running it into the pan.

A. R. PECHINEY, Salindres: *Method of preparing chlorates.* (Germ. P. 15,493, March 10, 1881). By the usual method of manufacturing potassium chlorate, decomposition of the product resulting from the action of chlorine upon milk of lime, by means of potassium chloride, a part of the potassium chloride remains dissolved in the calcium chloride mother liquor, after the crystallization of the potassium salt. In order to reduce this loss to a minimum the inventor removes the greater part of the calcium chloride from the mixture of calcium chlorate and chloride before adding the potassium chloride. This he effects by evaporating the liquid to 48° Be. and cooling, whereupon calcium chloride crystallizes, and can be removed, or by adding 3 molecules of lime for 1 molecule of calcium chloride whereby basic chlorides are formed, which can be separated from the liquid, or finally both operations may be employed consecutively.

C. LUCKOW, Deutz: *Electrolysis of zinc solutions for the purpose of extracting zinc from its ores.* (Germ. P. 14,256, April 20, 1880). Concentrated solutions of zinc salts are electrolyzed. The zinc is separated in grains, and the acids liberated are made to act upon crude or wasted zinc ores connected with the anode of the battery. Polarization of the electrodes is neutralized by chemical or mechanical means.

A. GURLT, Bonn: *Method of working up calamine residues and zinc waste by treatment with gaseous or liquid hydrochloric acid in combination with chlorides of the alkalies or alkaline earths.* (Germ. P. 15,012, Dec. 5, 1880, 1st additional patent to No. 8,116). The modification of the original patent consists in the employment of gaseous or liquid hydrochloric acid during the distillation of the zinc chloride, whereby the extraction of the zinc is facilitated.

P. MANHES, Lyons: *Manufacture of refined copper from copper mattes in Bessemer converters by the use of reducing agents.*

(Germ. P. 15,562, Aug. 20, 1880). Sulphurous copper ores are melted down in a pit furnace. The matte obtained is treated with air in a converter in the same manner as pig iron in the Bessemer process. The operation is ended when evolution of sulphurous acid has nearly ceased. The product is crude copper. To refine it, charcoal powder is blown in with the air. Modifications of the process are described for the treatment of mattes rich in iron or too deficient in sulphur to produce the required degree of fluidity.

MAX TSCHIRNER, San Francisco: *Improvements in the manufacture of explosive compounds*. (Germ. P. 15,508, Feb. 6, 1880). Picric acid, 1 part; tar, 1 part; and potassium chlorate, 5 parts.

I. M. LEWIN, Paris: *Use of cotton and dextrine for the preparation of a gelatinous nitro-glycerine*. (Germ. P. 15,073, Jan. 18, 1881). Purified and finely divided cotton is mixed with 5 parts of dextrine and heated under pressure in a closed vessel. The jelly thereby formed is dissolved in nitro-glycerine to the extent of 7 per cent., and forms a mass from which the nitro-glycerine will not drain. An explosive compound is formed by mixing this gelatinous nitro-glycerine with nitre and sawdust.

CHEMISCHE FABRIK AUF ACTIEN, Berlin: *Preparation of coloring matters from sulphosalicylic acid*. (Germ. P. 15,117, Dec. 10, 1880, and Germ. P. 15,889, Mar. 18, 1881). Sulphosalicylic acid is nitrated by means of nitric acid of sp. gr. 1.35. Stronger nitric acid eliminates the sulpho group with formation of various nitro-compounds. Nitro-sulphosalicylic acid and its soluble salts dye silk and wool yellow. The coloring power is augmented by the introduction of one atom of bromine. Excess of bromine also eliminates the sulpho group, forming dibromnitrosalicylic acid.

Sulphosalicylic acid also reacts with phenols to form coloring matters, yielding, for instance, with resorcin a brownish-red dye.

With diazo, diazoamido, and amidoazo compounds, sulphosalicylic acid produces red dyes.

In Patent No. 15,889, the method of preparation of the bromine substitution compounds of nitrosulphosalicylic and nitrosalicylic acids is claimed.

## UPON THE PREPARATION OF ARTIFICIAL INDIGO AND SOME OF ITS DERIVATIVES.

BY JAMES H. STEBBINS, JR., S. B.\*

It will be needless for me to tell you what indigo is, as you have probably all come across it, and know whence it is derived.

I will therefore only give you a graphic description of how this interesting body can be made synthetically; and in giving you this description, I can do no better than quote Baeyer's wonderful experiments :

Baeyer says : "The first thing I attempted to do was to remove the oxygen in isatine, and it will be remembered how this was accomplished, in 1865 and 1866, by the conversion of isatine into dioxindole, oxindole, and indole."

It was then tried to find an easier method for the production of indole, as Baeyer considered it to be the basis of the indigo group.

In 1868 Baeyer obtained it directly, by treating indigo with zinc dust ; and in 1869 he produced it synthetically from nitrocinnamic acid and its reduction product.

The following quotation may help to explain Baeyer's reasoning more thoroughly :

"If it is desired to make indole synthetically, it becomes necessary to introduce into benzol a diatomic carbon chain and a nitrogen atom, and then to connect the one with the other. The properties hereby necessitated will be found realized in nitrocinnamic acid, which has been deprived of carbonic acid, and the oxygen of the nitro group ; and, in fact, as we have already found, nitrocinnamic acid on being fused with potash produces indole."

Shortly after this, Kekule informed us that he had produced in a similar way isatine, from orthonitrophenylacetic acid. Baeyer and Suida then examined this compound more closely, and came to the conclusion that dioxindole and oxindole could be nothing else than the anhydrides of orthoamido-amygdaic acid and orthoamido-phenyl acetic acid. A synthetical examination soon after followed, and showed that oxindole by oxidation is converted into isatine ; which, however, was shortly afterwards produced by Claisen and Shadwell, by a new and convenient synthetical method.

In 1870, Baeyer and Emmerlin converted this isatine into indigo, and thus solved the problem he had so long been working at ; but, as the materials used (phosphorous trichloride, phosphorous, and acetyl chloride) were quite complicated, he strove to find a more simple method for accomplishing this end, and finally came to the

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\*Read at the January Conversazione.

conclusion that it would be better to first treat the isatine with phosphorous pentachloride, and then phosphorus, or some other reducing agent.

It was then tried to produce, in the same way, indole from oxindole; and, in fact, a body answering to the formula of a dichlorindole was obtained.

Without taking up any more of your time with the history of Baeyer's discoveries, I will now describe some of the products which may be converted into indigo blue.

#### ORTHONITROCINNAMAC ACID.

This acid may be easily prepared, according to Beilstein and Kullberg's process, and likewise the separation of the ortho from the paranitro compound by means of ether.

This acid bears a simple relation to indigo, as may be seen from the following formulæ:

*Nitrocinnamic Acid. Indigo.*



#### ORTHONITRODIBROMCINNAMIC ACID.

This acid is produced by treating the nitro compound with bromine under exclusion of sunlight.

The bromine compound crystallizes in short colorless needles, which melt at 180° C., under decomposition and formation of a small amount of indigo.

This body was found to have the formula  $C_9 H_7 (Br_2) NO_4 =$  orthonitrodibromcinnamic acid.

This body is soluble in caustic alkalies, without decomposition; which, however, by heat is converted into orthonitropropionic acid, and then into isatine. By heating an aqueous solution of the above acid with carbonate of soda, a small amount of crystallized indigo blue is the result.

#### ORTHONITROPHENYLPROPIOLIC ACID.



The bearing of orthonitrodibromcinnamic acid with alkalies differs considerably from the non-nitrated compound, inasmuch as HBr may readily be removed from the former, whereas from the latter it can only be removed by the energetic action of alcoholic potash to form propionic acid. The propionic acid is prepared usually from an aqueous solution of the dibrominated compound by treating with an excess of caustic soda solution.

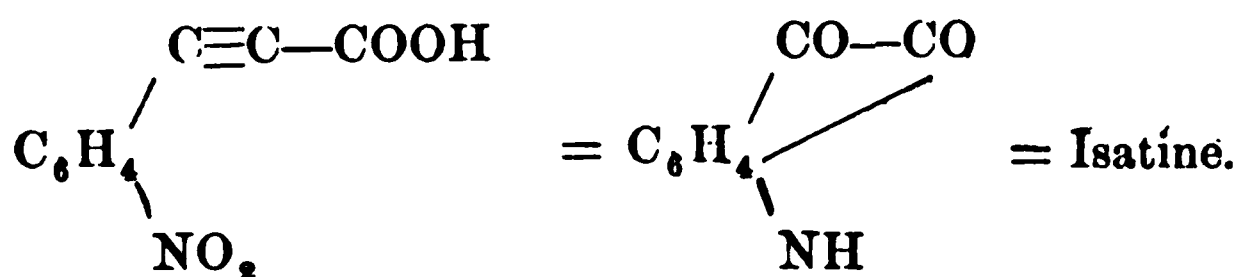
The compound is then thrown down by means of an acid, and dried. The orthonitrophenylpropionic acid so obtained appears as little colorless needles, melting at 155—160° C. with sudden decomposition.

#### ORTHONITROPHENYLACETYLEN.

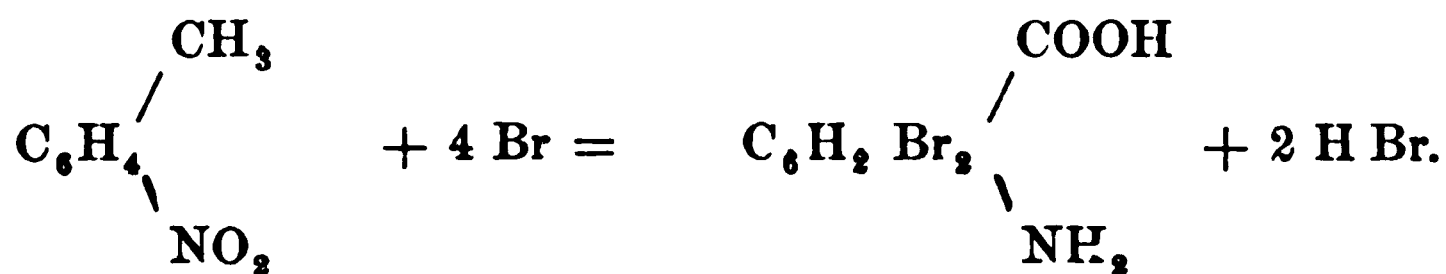
This compound may be obtained from orthonitrophenylpropionic acid, by boiling with water in an ascending cooler. It crystallizes from alcohol, in colorless needles, and melts at 81–82° C.

#### FORMATION OF ISATINE FROM ORTHONITROPHENYLPROPIOLIC ACID.

Isatine may be obtained in abundance by boiling a solution of orthonitrophenylpropionic acid with alkalis. The reaction that takes place here may be explained as follows :



In other words,  $\text{CO}_2$  is split off; at the same time a reduction of the  $\text{NO}_2$  group, and oxidation of the side chain takes place. This reaction is similar to the reaction which takes place when bromine is allowed to act on orthonitrotoluol, viz :

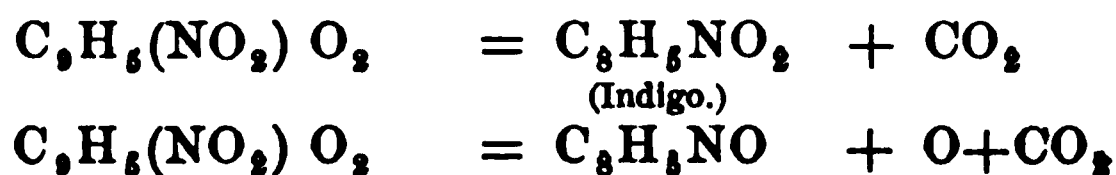


#### FORMATION OF INDIGO FROM ORTHONITROPHENYLPROPIOLIC ACID.

If a very dilute alkaline solution of the acid be heated to boiling, and a small piece of grape sugar is added thereto, the liquid first turns blue, and then follows a copious precipitation of small blue needles, with coppery red lustre. These needles are perfectly pure indigo-blue. The yield so obtained is about 40% of the propiolic acid used.

The formation of indigo-blue in this case takes place as follows :

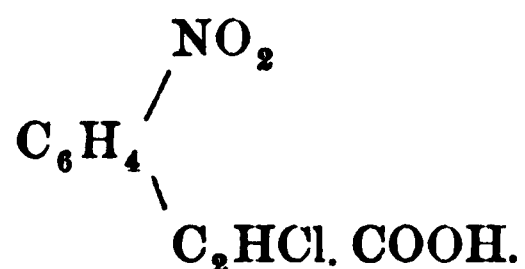
First the alkali, acting on propiolic acid, splits off  $\text{CO}_2$ , forming isatine, which, however, on being treated with a reducing agent as grape sugar, loses an atom of oxygen and forms indigo-blue, viz :



## ORTHONITROPHENYLCHLORLACTIC ACID.

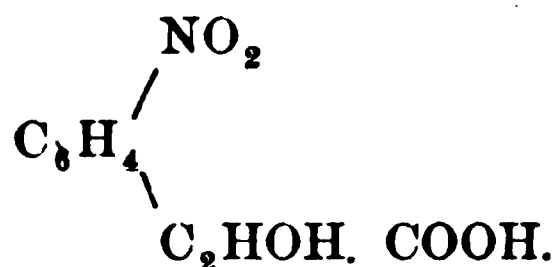
This acid may be easily prepared by passing a current of chlorine into an alkaline solution of orthonitrocinnamic acid. The mixture so produced is composed of orthonitrochlorstyrol, and orthonitrophenylchlorlactic acid, from which the latter may be separated by acidifying the mixture and then extracting the orthonitrophenylchlorlactic acid with ether. This compound appears as a white crystalline mass, and having a meltingpoint of  $119^{\circ}$ — $120^{\circ}$  C.

Its formula may be represented by



This compound on being treated with reducing agents, such as sodium amalgam, or sulphate of iron in an alkaline solution, forms indole.

## ORTHONITROPHENYLOXACRYLIC ACID.



This compound may easily be produced from orthonitrophenylchlorlactic acid, by treating with alcoholic potash. It crystallizes from hot water, in flat needles.

If heated slowly to  $110^{\circ}$  C. it melts, giving off  $\text{CO}_2$ , and forming a dark blue mass, which, on being treated with alcohol, dissolves with a brown color, in which small crystals of indigo-blue remain suspended.

Indigo-blue may also be derived from the above by heating its solution with phenol, but the yield of coloring matter so obtained is quite small.

The reaction taking place here may best be explained as follows:



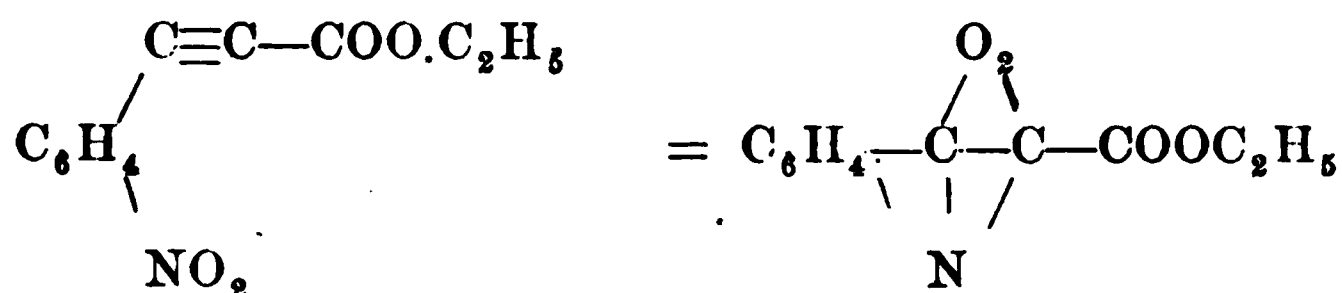
The ease with which orthonitrophenylpropionic acid is converted into indigo-blue, with such mild re-agents as grape sugar, appeared very strange to Baeyer, and in the following I will detail the experiments he made to arrive at a clear understanding of the above mentioned re-action.

By treating orthonitrophenylpropionic acid with strong  $\text{H}_2\text{SO}_4$ , at



the ordinary temperature, its isomer isatogenic acid ether is obtained, which crystallizes in yellow needles and melts at  $115^{\circ}$  C.

The formation of this compound may be explained by the following re-action :



A solution of nitrophenylpropionic acid in  $\text{H}_2\text{SO}_4$ , on being treated with reducing agents, forms a new blue dye-stuff, called indoine. Indoine is very similar to indigo blue but differs from it in that it dissolves in strong  $\text{H}_2\text{SO}_4$  with a blue color, which, on being heated, only forms a sulpho acid with great difficulty.

#### INDOXYLIC ACID ETHER.

This body is produced by the action of reducing agents, on isatogenic acid ether. It appears in the shape of thick prisms, and melts at  $120-121^{\circ}\text{C}$ .

It has the formula  $\text{C}_{10}\text{H}_{11}\text{NO}_3$ .

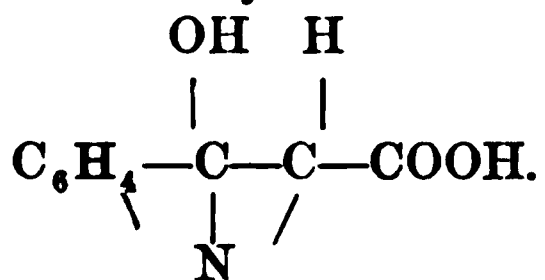
This compound on being rapidly heated forms a little indigo-blue; but if heated to  $100^{\circ}\text{C}$ . with strong  $\text{H}_2\text{SO}_4$  it passes over into indigo sulpho acid almost quantitatively.

On being fused with caustic soda, at a temperature of  $180^{\circ}$ , it forms indoxylic acid, which melts at  $160^{\circ}\text{C}$ .

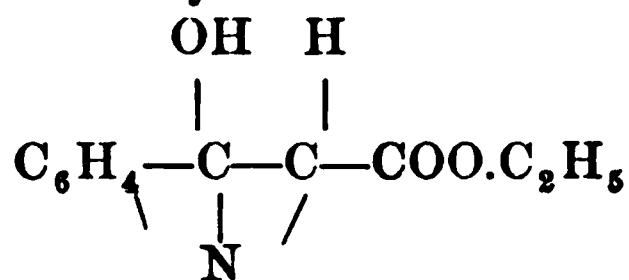
Ethyl indoxylic acid and nitrosoethylindoxylic acid have likewise been produced, but, I will not attempt to describe their formation.

The following formulæ will best represent the compounds just enumerated.

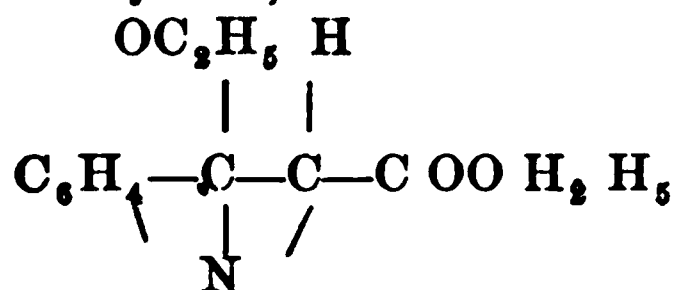
#### Indoxylic acid.



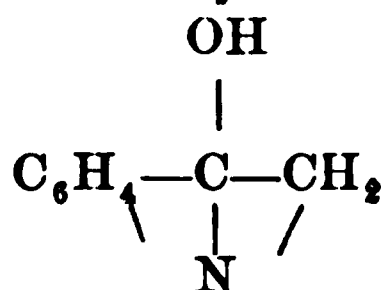
#### Indoxylic acid ether.

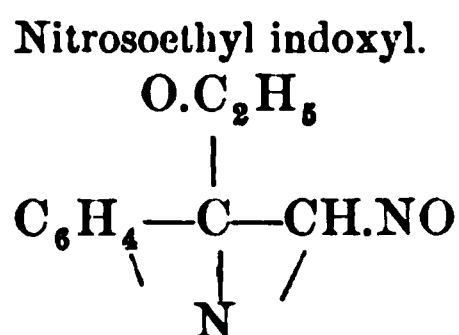
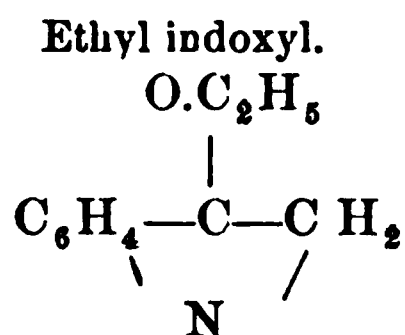


#### Ethylindoxylic acid ether.



#### Indoxyl.





Indoxylic acid on being heated as high as its melting point with  $\text{H}_2\text{SO}_4$ , gives off  $\text{CO}_2$ , and passes over into indoxyl.

If indoxyl be dissolved in dilute  $\text{KOH}$ . and exposed to the air, indigo-blue is formed, which falls out as a blue precipitate.

A mixture of indoxyl and orthonitrophenylpropionic acid, on being treated with  $\text{H}_2\text{SO}_4$  forms indoine.

Indoxyl and nitrophenylpropionic acid on being treated with  $\text{Na}_2\text{CO}_3$  are immediately converted into indigo-blue.

A mixture of indoxyl and isatine in alcoholic solution, on being treated with  $\text{Na}_2\text{CO}_3$  forms indirubine, which appears as brownish-red needles, with metallic lustre.

Thus far we have only considered the formation of indigo-blue from a theoretical point of view, and I will now try to describe the way it is made upon a large scale, or, rather, the manner in which it may be produced, and fixed in, or upon the fibre of materials at the same time.

The formation of indigo-blue within the fibre of materials to be printed, depends upon the facility with which orthonitrophenylpropionic acid is converted into indigo-blue, by the action of reducing agents.

The reducing agents more commonly used are grape sugar, and especially the xanthates of soda or potash.

In other words, to print a pretty blue upon calico, the orthonitrophenylpropionic acid is mixed up with a starch thickening, and xanthate of soda, and borax, and then printed in the usual way upon the calico. This is allowed to dry, and then heated to  $100^\circ\text{C}$ . for a certain length of time, which process develops the color within the fibre.

# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Adjourned meeting held Friday, May 19th, 1882.*

The meeting was called to order at 8:40 P. M., Dr. Chas. A. Doremus in the chair.

The first paper of the evening, "On the Examination of Some of the Methods for the Determination of Phosphorus in Iron Ores," by Dr. E. Waller was then read.

After giving the results obtained by methods suggested or recommended by Prof. Classen and M. Derome, Dr. Waller stated that he found a fusion of the pulverized ore with 2 to 3 times its weight of acid sulphate, finally using the blast lamp, afforded the phosphoric acid in the ore in a form soluble in dilute nitric acid, from which it could be separated by molybdate solution.

Dr. Prochazka spoke of the advantages of using Finkener's process (*Berichte Deut. Chem. Ges.* xi. 1638), in which most mineral acids can be used as solvents; and further stated that in applying this process, hydrochloric acid did not interfere with the precipitation of phosphoric acid. To get a complete and rapid precipitation, the solution should contain at least 20 per cent. of ammonium nitrate.

After some questions by Mr. Pitkin, the second paper, "Report of Progress in Industrial Chemistry," by A. A. Brenneman, was read by the Recording Secretary.

Prof. Brenneman described improvements in the manufacture of carbonate of soda, alum, &c., giving an interesting account of each.

After some remarks, Dr. Waller exhibited a piece of apparatus designed by Wm. Bettel, of England, for the estimation of nitrogen in water, beer, &c. The important feature of the apparatus consisted in a peculiarly constructed copper flask, in which by a continuous operation the ammonia is distilled off, and (indeed) a combustion of the residue with caustic soda is effected. It is claimed by the inventor to give good results.

After which the meeting adjourned.

JAMES H. STEBBINS, JR.,  
Recording Secretary.

## XV. AN EXAMINATION OF SOME OF THE METHODS FOR DETERMINING PHOSPHORUS IN IRON ORES.

BY E. WALLER, Ph. D.

The present investigation was undertaken with a view to obtain a more rapid method of determining phosphorus than those at present in use, by utilizing some suggestions which have recently been made.

Two lines of experiment were followed: That suggested by Prof. Classen (*Quantitative Analyse auf Electrolytischem Wege, Aachen* 1882), and that of M. P. Derome (*Comptes Rendus, LXXIX*, 925 translated in *Chem. News, XL*, 292).

For the purpose two limonites were taken, which by ordinary methods of analysis yielded as follows:

	"High"	"Low"
Fe	54.50	52.35
P	1.06	0.08

These had been pulverized and passed through an "80 mesh" sieve.

### ELECTROLYTICAL SEPARATION (CLASSEN).

In this the idea was to separate the iron by the use of the battery, thus incidentally determining the percentage of iron, and then, in the solution thus freed from the disturbing influence of such large quantities of iron (relative to the amount of phosphorus), to determine the phosphorus.

An experiment with an indefinite amount of ferric chloride showed that it took about two hours to deposit a little over 0.5 gm. of iron by the battery. The conditions, as specified by Prof. Classen, are that the solution—nearly neutralized—must receive the addition of a large excess of ammonium or potassium oxalate, and that the electrolysis shall be carried on in the solution heated to about 80° C. Three freshly charged Bunsen cells, of about 3 pints capacity each, were used. Prof. Classen states that enough oxalate must be used to form the double oxalate (of iron and ammonia or potassa) and then 3 or 4 gms. more of the oxalate must be added. The action of the battery converts the oxalate into bi-carbonate, and any deficiency of oxalate is soon perceived from the appearance of flocculent masses of ferric hydrate in the solution. It was found that considerably more oxalate in excess had to be used than the amount specified by Dr. Classen.

As so much oxalate was required it was not deemed possible—even with a platinum dish of over 250 c.c. capacity—to use more than 1 gm. of ore for the determination. The ore was fused with alkaline carbonates, dissolved in dilute  $\text{HCl}$ , and evaporated down to separate silica, taken up with dilute  $\text{HCl}$ , filtered, nearly neutralized, and the oxalate added. In the first experiment on the “High” ore only ammonium oxalate was used, and in this case, after a certain time, a flocculent precipitate of ferric hydrate formed, which refused to dissolve on addition of more alkaline oxalate, or to reduce to metallic iron, though the battery was allowed to act upon it all night. The solution, with precipitate, was therefore poured off and examined for phosphoric acid.

The substitution of potassium oxalate in part for ammonium oxalate, a proceeding suggested, though not insisted upon, by Prof. Classen, remedied this difficulty, though the amount of oxalate necessary was so great that the solution, when cold, was nearly solid with the interlocking crystals of the alkaline salts.

The action of the battery, aided by the temperature necessary, ( $80^{\circ}$  to  $100^{\circ}$  C.) kept the solution in such a lively state of effervescence that some of the solution was inevitably lost mechanically. When the solutions gave no reaction for iron they were poured off, the dishes rinsed with hot water, and finally with alcohol and ether. The deposited iron was not quite smooth, apparently containing granules of what was probably alumina hydrate or phosphate, which tenaciously adhered, and in some cases appeared to be partially covered with a coating of metallic iron. The solutions also were slightly turbid, apparently from the presence of alumina compounds. These solutions were evaporated down twice with concentrated nitric acid, then diluted and filtered into a molybdate solution containing 10 or more gms. of ammonium nitrate; the molybdate precipitate, after heating for some hours, filtered off, washed, dissolved in ammonia and the phosphoric acid separated by magnesia mixture. The results were :

	“ High ”	“ Low ”
Fe	56.01	50.45
P (Fe partly separated)	1.04	
P (Fe entirely separated)	0.988	0.089

Prof. Classen remarks (p. 29): “In presence of alumina the electrolytic method gives but little advantage in the determination of phosphoric acid. In this case a precipitation of that constituent in a separate portion with molybdate solution is to be preferred.”

The observation might obviously have been applied to the determination of iron also.

#### SULPHATE METHOD (DEROME).

M. Derome's method consists in fusing the mixture of ferric oxide, alumina and phosphoric acid, (precipitated by ammonia or basic acetate), with five to six times its weight of dry sodium sulphate over a blast lamp for 8 to 10 minutes, and then extracting with water. In the solution thus obtained it is asserted that the phosphoric acid can be directly precipitated with magnesia mixture. The ores were fused, &c., as above described, and the amount of filtrate from silica, representing 1 gm. of ore, precipitated with ammonia, washed, dried, ignited and fused as prescribed. To insure the change to sodium phosphate, the fusion was kept up for 15 minutes at the highest attainable heat of the blast lamp.

The water solution was found to contain small amounts of iron and manganese, possibly some other compounds, so that the addition of magnesia mixture gave a precipitate containing much beside pure magnesium ammonium phosphate, and it was found necessary to separate by molybdate solution before using the magnesia mixture. The precipitate, taken just as it was, after ignition, appeared to be too coarse to permit of the satisfactory action of the sulphate, so in one experiment it was pulverized finely before fusing. The results for phosphorus were :

	" High."	" Low."
Precipitate fused direct.....	0.669	0.028
"    pulverized and fused.....		0.058

Experiments were also made as to the effect of fusing the ore direct with the neutral sulphate. On account of the presence of small amounts of lime (under 1 per cent.), the mass after fusing was extracted with acid. Hot acetic acid took out much of the phosphoric acid which refused to dissolve in water, but interfered perceptibly with the precipitation by molybdate solution. Dilute nitric acid was found to dissolve what would dissolve out satisfactorily, the greater part of the iron oxide remaining undissolved in crystalline scales. The results were :

	High.	Low.
Ore fused with $\text{Na}_2\text{SO}_4$ .....	0.356	0.0194

The use of some acid sulphate was then tried; 3 gms. of each ore were fused with 3 gms.  $\text{NaHSO}_4$  at a low red heat until fumes ceased to come off ; then 3 gms.  $\text{Na}_2\text{SO}_4$  were added, and the whole

fused at the highest heat of the blast lamp for ten minutes. On cooling the mass was extracted with about 200 c.c. of water, to which was added 10 c.c. conc. nitric acid, and after heating for half an hour, at just short of boiling, the solution was filtered into molybdate solution into which some 10—15 gm. of crystals of ammonium nitrate had been thrown, &c., the phosphorus being finally weighed as the magnesium compound. (See result below). The neutral sulphate appeared to give but little advantage, and in this case the proportion of acid sulphate was evidently insufficient to completely disintegrate the ore. The experiment was therefore repeated, using three times as much  $\text{NaHSO}_4$  as ore; fusing for some time (one hour or a little over) at a low heat until fumes had almost ceased to come off, and then using the blast lamp for full ten minutes, extracting with dilute nitric acid, &c., as before. The results in the two last experiments were:

	High.	Low.
Ore fused with equal weights, $\text{NaHSO}_4$ and $\text{Na}_2\text{SO}_4$ ,	0.98	0.067
Ore fused with three times its weight, $\text{NaHSO}_4$ ,	1.05	0.083

This last mode of procedure, then, appears to be satisfactory for a rapid method of determining phosphorus in an iron ore. The amount of iron going into solution in the nitric acid is extremely small, so that that disturbing influence is removed. I propose making further experiments on the application of this method to titaniferous and other ores, and to bar iron and steel.

# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Room 1 University Building, N. Y., regular meeting June 3d, 1882.*

Dr. A. R. Leeds in the chair.

The minutes of the previous meeting were read, and after some corrections, were approved.

The minutes of the meeting of the Board of Directors on May 9, 1882, were then read.

The following gentlemen were then unanimously elected as regular members:

G. E. Bailey, proposed by M. Benjamin, W. Rupp and A. H. Elliott; Dr. Wm. Stratford, of the College of the city of New York, proposed by Chas. A. Doremus, Jas. H. Stebbins Jr., and A. R. Leeds; H. Edward Stockbridge, proposed by E. H. Goessmann, H. Endemann, and Jas. H. Stebbins Jr.; J. E. Mallett, Hoffman House, N. Y., proposed by C. A. Doremus, Jas. H. Stebbins Jr. and A. R. Leeds.

As associate member:

Y. Polledo, School of Mines, Columbia College, proposed by T. O'C. Sloane, Jas. H. Stebbins Jr. and A. H. Elliott.

The Librarian reported that Nos. 1 to 9 of the *Chemiker Zeitung*, and also some numbers of the *B. richte der Deutschen Chem. Ges.* were missing, and had been improperly removed from the library.

After which the following paper was read: On "Acetate of Lime, its manufacture and analysis," by Messrs. C. M. Stillwell and T. S. Gladding.

Dr. Waller remarked that the presence of acetic acid interferes decidedly with the precipitation of phosphoric acid by molybdate solution.

Mr. Stillwell replied that he overcame this difficulty by using large quantities of ammonium nitrate.

Dr. Grothe asked if the authors had noticed the presence of acetone in the products of the decomposition of acetate of lime by heat.

Mr. Stillwell said he had not noticed it particularly, since his attention had been confined simply to the loss of acetic acid.

Dr. Squibb remarked that it was not necessary to carbonize the wood, but that all the acetic acid could be obtained at a much



lower temperature, (about 160 to 200° C.) and the products then contain no acetone.

For the analysis of sodium acetate he uses a glass retort covered with copper gauze, and heats it on one side to prevent frothing and bumping. He uses 10 gms. of the acetate with 20 c.c. of water, and 10 c.c. sulphuric acid; after distilling off 10 to 15 c.c. of liquid, the distillation is interrupted,—about 20 c.c. of water are added to the retort, and 10–15 c.c. of liquid again distilled over. This is repeated a third time when all the acetic acid will have come over, without trouble from frothing or bumping during distillation.

Dr. Squibb further remarked that he used in his factory, retorts 20 feet long, 2 feet wide, and 10 feet deep, holding 2½ cords of wood, and heats them in a hot air bath. During the first twelve hours, only water is given off. In about 24 hours the acetic acid begins to distill over, the process being completed in 6 to 7 days for each retort.

Seasoned oak is preferred, and any admixture of softer woods diminishes the yield of acetic acid. Chestnut is notable in this respect.

Throughout the active heating, but more copiously toward the end, a gas, colorless and odorless (and, so far as tested, unflammable), comes over, having anæsthetic properties. This gas adheres to the wood, after the charge is cooled and drawn, and seems to be a reason why small vermin will not remain near it. If the charge is heated too long, smoke appears at the exit pipe and carbonization of the wood begins at the centre of the top of the charge, extending in V-shape toward the bottom. When once started this carbonization proceeds spontaneously without further application of heat. Indeed it is sometimes found difficult to check it, even by the liberal application of cold water to the outside of the retort.

In the course of the operation, when properly managed the charge shrinks one third of its volume. 4000 lbs. of wood yield about 2800 lbs. of residue. The residue retains all the appearances of the wood before distillation, only that it becomes walnut-colored and it has the same elementary composition as that of kiln-dried wood. It is brittle and not well adapted to construction, but forms a most excellent fuel for many purposes, especially for kindling anthracite coal. The distillate is neutralized with soda ash and distilled to about one-fortieth its volume. The first product is crude wood spirit. This is redistilled, and gives rectified wood

spirit, and lastly wood oils, which contain large quantities of furfural and no acetone. The wood oils are separated by passing the last portions of distillate into water. The rectified wood spirit contains about 80 % of methyl acetate, and when saponified gives a very pure methyl alcohol.

One cord of well-seasoned wood will afford 1200 to 1400 lbs. of liquid products. A cord of oak yields 60 to 70 lbs. of glacial acetic acid.

Dr. Squibb further remarked that there was no process commercially practicable for obtaining pure acetic acid from pyroligneous acid, or acetate of lime, but only from acetate of soda. Mr. Parker remarked that he had tried a process for making acetic acid by heating wood fibre with steam, under a pressure of 60 lbs. at 275° C. and confirmed Dr. Squibb's observations on the deterioration of the woody fibre, and also the impracticability of making pure acetic acid from pyroligneous acid.

Dr. Grothe remarked that in distilling acetone with bleaching powder, he had obtained chloroform simultaneously with calcium acetate.

Dr. Doremus brought up the question of giving reprints of papers to members. After some discussion Mr. Casamajor moved that "those members who desire reprints of their papers shall notify the committee on papers and publication, who may make arrangements to have them supplied at the expense of the authors." Carried.

After which the society adjourned to September 1st, 1882.

ARTHUR H. ELLIOTT,  
Rec. Sec. pro tem.

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## XVI. ACETATE OF LIME—ITS MANUFACTURE AND ANALYSIS.

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BY STILLWELL & GLADDING.

Acetic acid and its compounds hold a very important place in the arts and manufactures. They are extensively used in many branches of industry, as, calico-printing and dyeing, in the treatment of gums, in the manufacture of paints and varnishes, etc. Acetic acid is well known in the form of vinegar.

It may be formed in three ways: (a) by acetous fermentation, chiefly used for the making of vinegar; (b) by destructive distilla-

tion of wood, yielding crude pyroligneous acid and by further treatment, acetates of iron, lime, lead, soda, etc.; (c) by the distillation of various metallic acetates with sulphuric or hydrochloric acid, yielding the pure acid of commerce.

We will confine ourselves for the present to the second method of obtaining this acid—by the destructive distillation of wood:

“Wood consists essentially of woody fibre or cellulose, a small amount of mineral matter and sap and a variable amount of hygroscopic water. The woody fibre constitutes about 95 per cent. of dry wood, and is composed of, in 100 parts: carbon, 44.45; hydrogen, 6.17; oxygen, 49.38. The sap consists chiefly of water, holding organic and inorganic matters partly in solution and partly suspended. The inorganic constituents of the sap (the ash left after the incineration of the wood) are the same in all kinds of wood. In practice it is assumed that wood leaves about 1 per cent. of ash; but there is a difference for certain portions of the tree, the trunk yielding about 1.23 per cent. of ash, the branches and knotty parts 1.34 and 1.54, and the roots 2.27 parts of ash respectively.

Wood dried at 266° Fah.—at which temperature all the hygroscopic water is driven off—is composed of :

50 parts of carbon (inclusive of one part ash).

50 “ chemically combined water.”

In the process of dry distillation the hygroscopic moisture of the wood is first driven off. As the heat increases, acetic acid and water are formed by the decomposition of the wood, then follow the tar and volatile oils and at a still greater heat carbonic acid, carbonic oxide and marsh gas appear. The remainder of the carbon is left in the retort in the form of charcoal.

The following table from Wagner's Chemical Technology, shows at a glance the numerous and complex products which are formed during the destructive distillation of wood:

Wood.	{	1. Real wood. 2. Hygroscopic water.	{	a. Illuminating gas.	{ Acetylen. Carbonic oxide. Elayl. Carbonic acid. Benzol. Marsh gas. Naphthalin. (?) Hydrogen.
				b. Tar.	{ Benzol. Oxyphenic acid. Naphthalin. (?) Cresylic acid. Paraffin. Phlorylic acid. Reten. Empyreumatic resins. Carbolic acid. Creosote.
				c. Pyroligneous acid	{ Acetic acid. Aceton. Propionic acid. Wood spirit.
				d. Wood charcoal.	

Woods differ greatly in the amount of water which is present in them. The following table by Schuebler and Hartig shows the amount of water present in some freshly cut woods:

Hornbeam	contains	18.6	per cent.	of water.
Willow,	"	26.0	"	"
Ash,	"	28.7	"	"
Birch,	"	30.8	"	"
Oak,	"	34.7	"	"
Pine,	"	39.7	"	"
Red Beech,	"	39.7	"	"
Elm,	"	44.5	"	"
Larch,	"	48.6	"	"
White Poplar,	"	50.6	"	"

Different woods yield different results, hence the wood selected must be chosen with reference to the nature of the products desired. If the charcoal is to be used for making powder, alder, willow and dogwood are used ; if for heating, oak and beech ; if for pyroligneous acid alone, birch, thorn, and apple. Oak holds the first place and beech the second when we take into account all the products obtained—charcoal, acetic acid, naphtha, and tar.

The following table, by Stolze, shows the various products obtained from different woods :

336 lbs. wood.	Charcoal.	Acid Liquor.	Sp. Gr.	Dry Acetate of Lime.
Beech.....	84	180	1.029	25
Walnut.....	72	150	1.018	14½
Birch, cut 3 years...	70	120	1.031	13
Oak.....	91	190	1.022	24
Ash.....	90	190	1.024	22
Apple.....	70	200	1.017	18
Wych Elm.....	70	180	1.018	16
Maple.....	77	145	1.018	20

The average production of three works in Paris gave from 4,000 pounds of beech and oak : Charcoal, 1,014 lbs. ; Acid liquor, 2,335 lbs., sp. gr., 1.027, = dry acetate of lime, 416 lbs.; tar, 330 lbs.

When wood is distilled in close cylinders, the charcoal obtained is about 25 per cent. of the weight of the harder woods, as oak and beech, or about three-fifths of the total carbon present ; the liquid part is about 53 per cent., containing about one-fifth of the carbon; the gases contain the remaining one-fifth of the carbon. Of the

liquid products, acetic acid equals about 3 or 4 per cent. of the harder woods, the naphtha about 1 per cent. and the tar  $7\frac{1}{2}$  per cent.

The time occupied in firing a charge is from twelve to sixteen hours; the latter time is preferable, since a slow carbonization yields a larger product. The amount of charcoal obtained depends largely on the length of time occupied. The following table, by Karsten, gives the results of some experiments upon air-dried timber :

SPECIES OF WOOD USED.	CHARCOAL.	
	By quick charging.	By slow charging.
Young oak.....	16.54	25.60
Old ".....	15.91	25.71
Young beech.....	13.12	25.22
Old ".....	13.65	26.45
Young alder.....	14.45	25.65
Old ".....	15.30	25.65
Young birch.....	13.05	25.05
Old ".....	12.20	24.70
Young deal.....	14.25	25.25
Old ".....	14.05	25.00
Young fir.....	16.22	24.72
Old ".....	15.35	24.75
Young pine.....	15.52	26.07
Old ".....	13.75	25.95
Lime.....	13.30	24.60

According to an English authority, the yield which should be obtained can be approximately calculated from the following data. One ton of wood, delivered at the works, will yield  $2\frac{1}{2}$  to 3 cwt. of bark, and, if fairly dry, 5 cwt. best charcoal,  $1\frac{1}{2}$  to 2 cwt. of best brown acetate, or  $1\frac{1}{2}$  of grey,  $1\frac{3}{4}$  to 2 gallons of "miscible" naphtha at  $60^{\circ}$  over proof, or  $2\frac{1}{2}$  gallons of "solvent" naphtha at  $45^{\circ}$ .

Many improvements in the methods for wood distillation have been preposed from time to time, especially with reference to the utilization of wood in a finely divided state, such as spent residues from tanning and dyeing operations, saw-dust, &c. With properly constructed apparatus, the yield of acid is nearly as large as with fresh wood, but the charcoal, owing to its finely divided state, is almost worthless.

The use of hydrochloric acid for making a superior quality of acetate of lime is as follows: A clear solution of brown acetate is made and evaporated to one-half its bulk, removing the impurities

as they rise to the surface. When evaporated, hydrochloric acid is added until litmus paper is just reddened. The resinous products are thus separated and the volatile bodies driven off on further evaporation. About 5lbs. hydrochloric acid to every 41 gallons of liquid, after the naphtha has been expelled, are usually sufficient. The solution is then boiled down to dryness, giving a brown acetate of a remarkably pure quality.

This use of hydrochloric acid is not general in America. It has this disadvantage, that it leaves in the finished product a certain amount of chloride of calcium. When an acetate thus made is distilled with sulphuric acid to obtain the acetic acid the chlorine is carried over as hydrochloric acid and condenses with the acetic acid. We have tested many samples of American make of acetate of lime and find but very few which contain any chlorine at all; in every case where it existed in more than traces, the acetate had been made from shell-lime and hence the chlorine was present in the form of salt.

The lime used for making acetate should be as pure as possible and free from magnesia and silica. Both retard the slaking. The presence of 1 to 2 per cent. of magnesia is not specially injurious; at 10 per cent. the lime is perceptibly deteriorated and at 20 per cent. and upward it is too poor for use. Lime made from oyster shells is sometimes used. It contains more or less of salt, which is therefore carried through the various operations of manufacture and appears in the finished product. The salt may vary in amount from one per cent. to six or even more in the acetate of lime. Its presence is injurious when sulphuric acid is used for distillation as previously mentioned under the use of hydrochloric acid in the manufacture. It is also liable to cause serious mistakes in the chemical analysis of acetate of lime when analyzed by distillation, unless specially guarded against. We shall refer to this later on when we come to speak of methods of analysis. A sample of stone lime which gave an acetate of low strength, was found to contain 5.49 per cent. of magnesia; another lime giving an acetate of a better quality, was found to contain but 1.35 per cent. of magnesia.

Having thus glanced at the materials which enter into the manufacture of this important article of commerce, we are prepared to discuss the different kinds of acetate of lime now manufactured and to call special attention to a few points which have much to do with its successful production.

Some years ago, three kinds of acetate of lime were made, the

black, the brown and the grey: but with improved methods of manufacture the black acetate has disappeared and but two kinds, the brown and the grey are now in use. The black acetate contained on an average about ten per cent. less acetate of lime than the brown.

In order to show the composition of representative samples of the brown and the grey acetates of lime, we give below two analyses, each of which is an average of twenty-six samples, taken at random from samples analyzed by us during the last three months. The reaction of both samples is alkaline, from the presence of caustic lime.

	Brown.	Grey.
Moisture, at 100° C.,.....	6.40	4.07
Insoluble, sand, &c.,.....	1.13	0.87
*Lime.....	23.95	29.29
Magnesia.....	0.62	0.15
†Acetic acid, anhydrous, .....	42.42	53.34
Tar, coloring matter, &c.....	25.48	12.28
	<hr/> 100.00 <hr/>	<hr/> 100.00 <hr/>

\*By *English Commercial Test*.

Equiv. to acetate of lime.....	67.37	82.64
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†By *Distillation*.

Equiv. to Glacial acid....	49.51	62.32
“ “ Acetate of lime.....	65.19	82.05

The following tables of analyses include all the samples which have been analyzed by us during the last two years, ending April 30, 1882. The average of the brown acetates would be slightly increased if it included samples of a few makes of high-grade brown acetate; but as these lots are usually sold without analysis they do not enter into the average given here. The analyses given below are a fair average of the general class of brown and grey acetates of lime now in the market.

**Brown Acetate of Lime ;—By *English Commercial Test*.**

No. of samples.	Moisture at 100° C.	Acetate of Lime.	Acetate of Lime (on dry basis.)
179	7.13	67.53	72.71

**Grey Acetate of Lime ;—By *English Commercial Test*.**

No. of samples.	Moisture at 100° C.	Acetate of Lime.	Acetate of Lime (on dry basis.)
84	5.33	81.01	85.57

**Grey Acetate of Lime ;—By *Distillation*.**

No. of samples.	Moisture at 100° C.	Glacial Acid.	Acetate of Lime.	Glacial Acid (on dry basis.)	Acetate of Lime (on dry basis.)
22	6.22	62.60	82.42	66.75	87.88

This table of grey acetates, analyzed by distillation, is composed of exceptionally high-grade samples. They are very light in color, containing but little tar and yielding an amount of acetate of lime above the general average of the grey limes quoted above. By the "English Commercial Test" the average of these twenty-two samples would show about 89.00 per cent. acetate of lime on dry basis.

The presence of *chlorine*,—either as *chloride of calcium* from the use of hydrochloric acid in the manufacture, or as *chloride of sodium* from the use of shell-lime—is not usual in American acetates of lime. Out of twelve samples, taken at random from nearly sixty samples analyzed by us during the last three months, ten samples showed no trace of chlorine, one contained a trace only and one had about two per cent. of chlorine. Both the latter samples were made from shell-lime.

In order to make an acetate of lime of as high a grade as possible, three results should be sought :

First : The removal of the *tar*.

Second : The finished product should contain as little *water* as possible.

Third : An undue *elevation of temperature* during the drying of the acetate should be most carefully avoided.

In an article which contains so large a percentage of a valuable ingredient as does acetate of lime, the presence of even one per cent. of impurity causes a loss of 0.7 to 0.9 per cent. acetate of lime ; that is, the one per cent. of useless matter, be it insoluble matter, tar or water, takes the place which might be occupied by valuable acetate of lime.

For example : Two lots of brown acetate of lime are taken, each containing originally 9 per cent. of water and 66 per cent. of acetate of lime. One lot remains unchanged ; the second, by exposure, dries so that it contains 6 per cent. of water. The acetate of lime is therefore raised to 68.17 per cent. The total weight of the second lot would be less than it was at first, but the amount of acetate of lime in one hundred pounds, that is, stated as a percentage would be raised to 68.17. Both lots taken each as a whole would contain exactly the same total amount of acetate of lime. One ton of 2,000 lbs. weighed out from the first lot, which remained unchanged, would contain 1320 lbs. acetate of lime ; but one ton of 2,000 lbs. weighed out from the second lot, which had dried, would contain 1363.4 lbs. acetate of lime.



So of two lots of grey acetate of lime, each containing at first 9 per cent. water and 80 per cent. acetate of lime. By drying to 6 per cent. water the second lot would contain 82.63 per cent. acetate of lime in each one hundred pounds of the dried-out acetate.

Since acetate of lime when dry rapidly absorbs moisture, a sample containing only 2 or 3 per cent. of water might absorb as much more by exposure in a damp place, and the amount of acetate of lime would be proportionally diminished when reckoned into percentage on one hundred pounds. The total weight of the lot would be increased, but the actual amount of acetate of lime in each one hundred pounds of the dampened lot would be less than in one hundred pounds of the lot when first made.

This brings us to the third requisite needed for a high-grade article, the *Drying of the acetate of lime*. It is here that care is indeed needed. A rise of temperature of only 50° Fah. above the safety point begins at once to decompose the acetate of lime and the product rapidly loses in strength with further increase of temperature. We have been unable to find any special reference to this important point in the manufacture of this article. It is indeed stated in books that great care is needed in the drying and that the acetate is easily injured by heat, being decomposed at about 460° Fah. But we have met with no record showing the rate of decomposition produced by length of time or increase of temperature. We have made a long series of experiments on this point, because of its importance, in connection especially, with the manufacture of acetate of lime. We sought to ascertain the temperature at which the decomposition begins, and also to see what influence, if any, the impurities possess in hastening such decomposition. We used for this purpose a so-called chemically pure, white acetate of lime,—which had lost by efflorescence some of its water of crystallization, a commercial brown acetate of lime—the average of twenty-six samples, the full analysis of which has been already given, and a commercial grey acetate. An analysis, by distillation, was first made. Then weighed quantities of the samples were submitted to heat for different lengths of time as detailed below, and the residues were then distilled and the acetic acid determined. The results are calculated to a dry basis for better comparison.

A preliminary trial with a sample of acetate of soda, showed that it was not at all decomposed by an exposure for one hour at 550° Fah.

ACETATE OF LIME—PURE.

	Glacial Acid.	Acetate of Lime.
Sample originally contained,	74.53%	98.13%
Heated for 1 hour, at 500° Fah.	74.35	97.89
“ “ “ “ 550° “	73.32	96.54
“ “ “ “ 600° “	72.08	94.90

The acetate after heating at the high temperatures above given, was strongly alkaline, from caustic lime. At 500° Fah. no change took place in the color; at 550° Fah. it was in places very slightly browned; at 600° Fah. it was but little darker in color than at 550° Fah. Chemically pure acetate of lime contains 75.95 per cent. of glacial acid, on a dry basis.

	BROWN ACETATE.		GREY ACETATE.	
	Glacial Acid. per cent.	Acetate of Lime. per cent.	Glacial Acid. per cent.	Acetate of Lime. per cent.
Sample originally contained,	52.89	69.64	67.09	88.33
Heated for ½ hour, at 400° Fah.,	52.77	69.48	67.09	88.33
“ “ “ “ 450° “	50.15	66.03	66.17	87.12
“ “ “ “ 500° “	50.15	66.03	64.86	85.40
“ “ “ “ 550° “	48.83	64.29	63.40	83.47
Heated for 1 hour, at 400° Fah.,	51.01	67.16	67.09	88.33
“ “ “ “ 450° “	50.16	66.04	65.89	86.75
“ “ “ “ 500° “	49.65	65.37	64.09	84.38
“ “ “ “ 550° “	48.83	64.29	63.09	83.07
“ “ “ “ 600° “	—	—	61.94	81.55

We have here, we think, a clue to at least one of the causes which sometimes yield an acetate of lime far lower in strength than was expected by the manufacturer. We see by the above table that the greater part of the loss occurs during the first half hour of exposure at 500° Fah., that it is three times as great in the brown acetate as in the grey, and that for every additional 50° Fah., the loss will average about 1.40 per cent. of acetate of lime. In a substance so easily decomposed by a high temperature as is acetate of lime, we cannot expect that the results obtained will be in exact ratio to the increase of time or temperature. The figures are given just as obtained from the analyses. Several of them, over 500° Fah. are the results of duplicates, the greatest difference being one half of one per cent. for an hour's heating.

The limit of safety is 400° Fah., for the grey acetate, and 400° Fah. for one half an hour for the brown acetate. This temperature.

will brown white paper by an exposure of fifteen or twenty minutes. An increase of fifty degrees up to 450° Fah., would not be readily noticed by the operative, but its results would surely be found in the poorer quality of the article produced.

When brown or grey acetates of lime are distilled in a current of steam the distillate is always more or less colored. But after exposure to a temperature of 450–500° Fah. for half an hour or so, the distillate is perfectly colorless. The tarry matters are no longer carried over, but this advantage is gained at a loss of a certain part of the acetate by decomposition, as shown above.

Many samples of acetate we analyze contain pieces or crusts which appear to have been melted or partly burned; some lots contain a large amount of such pieces and their presence must diminish to a certain extent the percentage of real acetate present. A sample of the grey acetate referred to in the table above, was heated for an instant until partly melted and the glacial acid was found to be 65.46 per cent. a reduction of 1.63 per cent., equivalent to 2.14 per cent. acetate of lime.

#### SAMPLING.

In drawing samples for analysis it is important to obtain a fair average of the lot. One bag in every ten is the usual number sampled; if the lot is small, one bag in every five or six is sampled, care being taken to obtain a fair proportion of both large and small pieces. The samples are taken from the bags by means of a large cylindrical tryer and put at once into a rubber bag to protect them from change, either by absorbing or losing moisture. The average sample thus obtained is taken to the laboratory and prepared for analysis by thorough mixing and sifting until the final average is reached. The moisture of the sample, as drawn, is determined and is reported in the certificate of analysis.

*The final average prepared for analysis may have lost or gained moisture during the operations of grinding, &c., hence it is necessary to make a correction of the analytical results obtained, in order to report the correct amount of acetate of lime present.*

For example: a sample of brown acetate prepared for analysis contained 7.04 per cent. moisture, and 70.33 per cent. acetate of lime. But the sample as drawn from the bags contained 9.74 per cent. water; hence the result of 70.33 per cent. is corrected to 68.28 per cent. based as the 9.74 per cent. of water actually present in the goods. A sample giving 66.86 per cent. acetate of lime, with

7.00 per cent. of water in the prepared sample was corrected to 67.66 per cent. based on 5.90 per cent. water present in the original sample. Some samples containing large amounts of tar or moisture need a preliminary drying of the sample selected for analysis: hence the correction to be applied is much larger than is usual, where the samples are easily ground and sifted.

It sometimes happens that samples are received for analysis, which come in paper boxes or wrappers. Such a sample will very seldom represent the actual condition of a lot of acetate as shipped from the factory. The paper box or wrapper offers but little obstruction to a loss of moisture by drying. Hence the sample received is very likely to give a much better result than will be obtained when the goods are received, and sampled from the bags for analysis.

*The amount of moisture present should always be stated in the certificate of analysis of acetate of lime, because it is an important factor, showing the condition of the goods at the time of sampling. This determination of moisture must be made on the sample as drawn, before it has been subjected to any treatment which will at all affect the amount of moisture present.*

#### METHODS OF ANALYSIS.

##### 1.—*Based on the Amount of Soluble Lime salts present.*

The value of an acetate of lime depends entirely on the amount of glacial acetic acid present. Two methods of analysis are in use at the present time; one determines the amount of lime salts soluble in water, and by calculation the amount of lime so found is converted into acetate of lime, or glacial acid, as the case may be. This method is based on the supposition that all the soluble lime salts present are acetates; but this is not so. Acetates of lime almost invariably contain caustic lime in slight amount, and if the lime has been overheated it is present in still greater quantity. Again, organic salts of lime are always present. With improved methods of manufacture, the amount of these has diminished of late years, and the difference between the amounts of acetate of lime found by distillation of the acetic acid, and that found from the amount of soluble lime is much less than formerly. The better the sample of acetate, the less is the difference between the results given by the two methods.

In the year 1872 (see *American Chemist*, vol. II, p. 324, and vol. III, p. 8), this question of methods of analysis came up. It is un-

necessary to state here the objections raised by certain English chemists against the process of distillation, which was advocated by some American chemists, since the objectors to a distillatory process had no good foundation upon which to rest. But as the general custom of the trade was at that time based upon the method of analysis by means of the soluble lime salts present, we have before and since that date used the term "*English Commercial Test*," to designate analyses made in this manner, and so state the results on our reports of analysis. Whenever an analysis is made by distillation it is so stated.

2.—*Based on the Distillation of the Acetic Acid.*

Three acids may be used in the process of distillation—hydrochloric, sulphuric or phosphoric.

*a.*—When *hydrochloric acid* is used, a part of it is carried over with the acetic acid and must be estimated and a correction made. It possesses these two advantages: that it does not act upon the organic matter present, and that the solution of chloride of calcium will permit of the distillation being carried to a low point without danger of error. The distillation is made in a retort connected with a condenser, and the total acids present in the condensed liquid are estimated, and the proper correction applied for the amount of hydrochloric acid found to be present.

*b.*—The use of *Sulphuric acid* has three disadvantages.

*First.*—As it becomes concentrated in the retort during the process of distillation, it acts upon the organic matter present, forming sulphurous acid, which is carried over with the acetic acid and included in the estimation of the total acid power of the distillate. Thus the amount present would be calculated as acetic acid, and the result thereby increased unduly. To prevent such action, recourse is had to distillation in a current of steam. (See *American Chemist*, vol. vi. p; 294.)

*Second.*—The sulphate of lime formed by the addition of sulphuric acid to the solution of the acetate of lime, is troublesome on account of the bumping which takes place during the distillation. This is partly prevented by the use of a current of steam.

*Third.*—When a sample of acetate of lime contains *chloride of calcium* or *chloride of sodium*, the chlorine is carried over in the form of hydrochloric acid and neutralizes its equivalent amount of the standard soda, used to receive the acid distillate. The amount thus distilled must be estimated and the correction made. To prevent this, the chlorine present is precipitated by the addition of

sulphate of silver to the solution of acetate before distillation begins.

*c. Phosphoric Acid* is the best acid for use in the process of estimating acetic acid by distillation. It has three advantages.

*First.*—It does not act on the organic matter.

*Second.*—During the distillation, the liquid in the retort is not suffered to fall below 15 c.c. in bulk. Under such circumstances, phosphoric acid does not decompose any chlorides of calcium or sodium which may be present in the acetate, thus requiring no addition of sulphate of silver. Only the merest trace of chlorine is carried over, as shown by experiments with a sample of acetate containing nearly 5 per cent. of chloride of sodium and also by trial on a known amount of pure chloride of sodium. This unexpected result merits attention, since in this respect phosphoric acid differs so decidedly from sulphuric acid.

*Third.*—It forms a perfectly clear solution with the acetate of lime. By the use of a large amount of glacial phosphoric acid, at least five times the weight of acetate of lime taken for analysis, the phosphate of lime first formed is redissolved to a clear solution. The distillation proceeds quietly and uniformly without bumping and the amount of liquid in the retort is easily kept at any desired point. The use of a current of steam is necessary both for ease of manipulation and accuracy of results.

After a thorough trial of hydrochloric and sulphuric acids, we discarded both in favor of phosphoric acid and for some years past have used it to our full satisfaction. It is important that the phosphoric acid used should contain no nitric or other volatile acids, which would increase the results obtained above the truth. Each new lot of phosphoric acid should be examined for such impurities before use. If it be suspected that any phosphoric acid has been carried over during the distillation, it is easily detected in the distillate by the use of molybdate of ammonia.

The process of distillation, if carefully and intelligently done, is very accurate. Duplicates made by this method agree with each other fully as closely as do those made by the estimation of the soluble lime. For example: In January last, a sample of grey acetate was analyzed by distillation in our laboratory, with a result of 78.22 per cent. acetate of lime. Three months later, the same sample was reanalyzed by another man in our laboratory, using fresh standard solutions, and 78.20 per cent. of acetate was obtained. This sample contained 3.16 per cent. of common salt,

shell-lime having been used in its manufacture. See also the table of analysis quoted above under "Drying of the Acetate of Lime." With care and experience in this method of analysis by distillation, it is not difficult to obtain duplicates which agree to two tenths of one per cent. of acetate of lime; usually the difference is less than that. A sample of grey acetate analyzed recently, was distilled with sulphuric acid and a duplicate with phosphoric acid. The results agreed exactly; the latter distillation, however, requiring less attention than the former, for reasons given above under processes of distillation.

The indicator used for titration is a few drops of a solution of phenol-phthalein—one gramme in 250 c.c. of a mixture of equal parts of water and alcohol. We have found this indicator far preferable to either litmus or cochineal; of course, whichever indicator is used, the same must be used both for the standardizing and the actual analysis.

To show the need of some uniform and accurate method of analysis, we refer to a note in Allen's *Commercial Organic Analysis*, vol. I. p. 205. He finds from experiments made in his own laboratory on the *same sample* of acetate of lime, that results were obtained varying from 47.4 per cent. to 57.6 per cent. of acetic acid. In our own practice for many years previous to the publication of this book we found differences fully as great between results by distillation and by the various commercial processes; but, as we said before, at the present time the differences are not nearly so great as formerly.

In conclusion we wish to lay strong emphasis upon the facts, that, inasmuch as the process of distillation is the only one which gives the real amount of glacial acid present, and, inasmuch as it is an imitation of the actual manufacturing process for obtaining acetic acid from its acetates, it is the most reliable and should be adopted. This position we have maintained for many years, and we notice that, especially in the case of grey acetate, buyers are more and more insisting on the test by distillation. The price should be based on the unit of glacial acid, just as in fertilizing materials, the prices are based upon the units of phosphoric acid, nitrogen and potash.



## FOREIGN PATENTS.

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FARBWERKE, successor to MEISTER LUCIUS & BRUNING, Hoechst: *Method of preparing coloring matters by the action of the disulphonic acids of B-naphthol upon the diazo compounds of aromatic acids.* (Germ. P. 15,250, Jan. 22, 1881.) Red dyes result from the action of the two disulphonic acids of B-naphthol upon the diazo-compounds of ethylic and methylic ethers of aromatic acids. The methylic and ethylic ethers of para-diazocinnamic acid yield red, those of diazo-a-naphthoic acid bluish red, and those of diazo-B-naphthoic acid claret red coloring matters.

O. N. WITT, Muelhausen: *Method of preparing coloring matters by the action of nitroso-compounds or chloroquinoneimides upon aromatic metadiamines.* (Germ. P. 15,272, Nov. 6, 1880.) By the action of the nitrosoderivatives of tertiary aromatic amines, or of nitrosophenols upon metadiamines, particularly upon phenylene and toluylene-diamine coloring matters result which upon oxidation yield new coloring matters. Instead of nitroso-compounds the so-called quinonechlorimides, obtained by allowing solution of chloride of lime to react upon hydrochloride of amidophenol or upon the chlorhydrates of paradiamines, may be employed. These yield blue and violet coloring matters which change to red upon boiling.

R. IRVINE, Royston Glanton: *Improvements in the manufacture of lubricants.* (Germ. P. 15,397, Nov. 24, 1880).

F. HEEREN, Hanover; *Method and instrument for testing milk.* (Germ. P. 15,681, Feb. 22, 1881). A disc upon which are six radial panels of different shades, the colors of which correspond to the appearance of milk of different degrees of fatness. There is also a dark central panel. Upon this a thin layer of the milk to be tested is produced. The panel corresponding in color with this layer shows the degree of fatness of the milk.

C. SCHEIBLER, Berlin: *Method of preparing saccharate of strontium from molasses and syrup.* (Germ. P. 15,385, July 24, 1880). Tribasic saccharate of strontium separates at a boiling heat from the above liquid, and is purified by washing with hot water. At a lower temperature the strontium saccharate so obtained is decomposed into a less basic saccharate and free strontium hydroxide. The former is used for defecating beet juice and similar liquids.

KUNHEIM & Co., Berlin: *Method of removing gypsum from solution of glucose obtained by the aid of sulphuric acid.* (Germ. P. 15,076, Jan. 29, 1881). Barium oxalate or phosphate is added to the solution after neutralization of the sulphuric acid with chalk. Instead of the above, any other barium salt the acid of which forms an insoluble compound with lime may be used.



P. HOSEMANN, Berlin: *Sizing for woven goods, wall paper, calico printing, felting &c.* (Germ. P. 15,251, Feb. 1, 1881). A size unalterable, odorless and neutral is prepared by mixing solution of calcium chloride, potato starch, water, rosin dissolved in potash and soda lye, and heating to 140–170°F. A mixture of "cream of tartar preparation," sulphuric acid and solution of aluminium sulphate stirred up in water, is then added. Finally solution of zinc chloride, water, phenol and nitrobenzole. Some of the above substances may be left out or others substituted to suit the purpose for which the sizing is intended.

H. SCHMIDT, Frankfort on M: *Method of preparing nitrobenzaldehyde from nitrobenzylchloride with the aid of metallic oxides.* (Germ. P. 15,881, Feby. 20, 1881). Paranitrobenzylchloride is heated to 200–250°C. with 1–2 parts oxide of copper or peroxide of lead. Besides the above mentioned substance, nitrobenzylbromide, sulphide and hydrosulphide can be converted into nitrobenzaldehyde.

A. P. DUBRUNFANT, Paris: *Manufacture of maltose.* (Engl. P. 1,048, March 11, 1881). Extract of malt is added to a dough of starch and warm water. The mixture is kept at a high temperature until the dextrine is all converted into maltose. The resulting liquid is clarified, filtered and evaporated *in vacuo*.

B. ROEBER, Dressle: *Disinfection and clarification of sewage and waste waters from industrial establishments.* (Germ. P. 15,392. April 25, 1879). Addition of lime and tar. Waters difficult to clarify receive besides the above a quantity of magnesium chloride.

C. V. CLOLUS, Paris: *Obtaining substances from soap boilers' waters.* (Eng. P. 681, Feb. 16, 1881). Neutralization with HCl evaporation to 32° Bé. to separate salt, and treatment of the liquid portion with hot air to remove water from the glycerine.

H. STIER, Gerstewitz: *Arrangement for rendering the hot waste gases from coke furnaces suitable for certain chemical purposes.* (Germ. P. 15,683, May 23, 1880). The heat of the gases is used for heating a cylinder containing coke in which water gas is generated. The latter, together with part of the above furnace gases, are employed in heating soda furnaces, etc.

A. ROSSI and L. BECKWITH, New York: *Method of generating cold.* (Germ. P. 15,151, Jan. 18, 1881). An ammonia ice machine in which the ammonia is dissolved in glycerine.

J. S. BARFF, London: *Preservative for organic matters.* (Engl. P. 1,335, March 25, 1881). Use of boracic acid dissolved with the aid of heat in glycerin.

E. CHESNAY, Paris: *Depilatory for hides.* (Germ. P. 15,736, Nov. 6, 1880). Solution of ammonium sulphite.

W. WOLTERS, Kalk, near Cologne: *Method of applying an*

*acid-proof coating to iron vessels used for distilling sulphuric acid.* (Germ. P. 15,639, Nov. 11, 1880). By heating a mixture of pyrosulphates of the alkalies and concentrated sulphuric acid in iron vessels, the latter are said to become coated with sulphide of iron, and to be acid-proof at the distilling temperature of sulphuric acid provided the acid sodium sulphate be left in the vessels during the distillation.

E. SOLVAY, Brussels: *Method of calcining the sodium bicarbonate obtained in the ammonia soda process.* (Germ. P. 16,131, May 10, 1881). Adds a certain quantity of calcined soda to the bicarbonate to prevent the latter from becoming pasty in the calcining apparatus.

E. SOLVAY, Brussels: *Method of utilizing native basic phosphates by employing them in the manufacture of soda and potash by the ammonia soda process.* (Germ. P. 16,229, April 15, 1881). The phosphates are concentrated in the following manner: They are first pulverized and washed in order to separate part of the lighter calcium carbonate. The powder is afterwards calcined whereby the lime not combined with phosphoric acid becomes causticized, it is then employed in decomposing the ammonium chloride resulting from the ammonia soda process. Calcium chloride dissolves out and calcium phosphate containing but little excess of lime remains.

H. KOECHLIN, LOERRACH AND O. N. WITT, Muelhausen: *Preparation of blue and violet dyes.* (Germ. P. 15,915, Mar. 19, 1881.) The coloring matters can be obtained by two different methods.

I. The nitroso derivatives of tertiary aromatic amines or phenols, as well as the so-called chlorquinoneimides, yield coloring matters if allowed to remain in contact with alkaline or ammoniacal solutions of phenols at the ordinary temperature. Their formation can be effected immediately if the re-action be initiated by the addition of reducing agents, as zinc dust, stannous oxide or grape sugar.

II. The same coloring matters are formed if weakly alkaline, neutral or weakly (preferably acetic) acid mixtures of phenols, with para-amido bodies of phenols, primary, secondary or tertiary aromatic amines be treated with oxidizing agents. As such atmospheric oxygen, chromates, ferricyanides, permanganates, etc., may be employed.

Instead of preparing the coloring matters they may be produced directly upon the fibre, a proceeding which possesses certain advantages in calico printing. Three processes for doing this are described in the patent. (See also this Journal vol. IV).

L. WOLLHEIM, Vienna: *Method of obtaining pure caustic alkalies by means of electrolysis.* (Germ. P. 16,126, Apr. 13, 1881.) A diaphragm divides the decomposing vessel into two chambers. In the one is placed the negative, in the other the positive electrode. At starting a solution of the alkali to be prepared is poured into

the former, into the latter a solution of the salt to be decomposed, carnallite for instance. If the electric current does not exceed a certain strength a continuous flow of caustic potash solution, uncontaminated with salt solution can be obtained.

A. HEGENER, Cologne : *Improvements in the fractionated distillation of coal.* (Germ. P. 14,889, Oct. 17, 1880.) The object of this invention is to obtain illuminating gas and fuel gas from the same charge of coal, the latter gas being chiefly evolved at the beginning and towards the end of the distillation. For this purpose the retort furnaces are provided with two separate mains and the dip pipes of both furnished with valves.

C. A. BURGHARDT, Manchester : *Treatment of animal and vegetable fats for the manufacture of soap.* (Eng. P. 5,191, Dec. 11, 1880.) The crude fatty matters are heated to a 130—180° in a retort by means of high pressure steam. As soon as vapors begin to form, a stream of cold or warm air is blown upon the surface of the fatty matters. The distilling fatty acids are absorbed by water or caustic lye.

H. P. HOSEMAN, Berlin : *Finish for woven fabrics.* (Germ. P. 16,110, Nov. 15, 1880.) The fabrics are passed through a solution of silk, wool or feather-down in caustic soda, then through a bath of sulphuric acid and finally through water.

ACTIEN—GESELLSCHAFT FÜR ANILINFABRIKATION, Berlin : *Manufacture of orange red, red and brown—red azo dyes from methylnaphthalene.* (Germ. P. 15,649, Feb. 18, 1881.) That part of coal tar which boils between 225° and 250° C. consists essentially of methyl and dimethylnaphthalene. From these, by nitration, conversion into the sulphonic acids and reduction amidomethyl (or, as the case may be, dimethyl) naphthalenesulphonic acid is prepared. The diazo compound of this acid yields coloring matters with betanaphthol or its sulphonic acids.

Or methylnaphthalene is transformed into the sulphonic acid and subsequently by fusion with potassa into methylnaphthol. This body yields with diazosulphanilic acid, or with diazonaphthalenesulphonic acid, or with amidoazobenzenedisulphonic acid yellowish red dyes; its sulphonic acid yields red dyes with the diazo-hydrocarbons.

ACTIEN GESELLSCHAFT "VIEILLE MONTAGNE," Angleur near Liege : *Manufacture of zinc white from solution of zinc chloride or sulphate.* (Germ. P. 15,249, Dec. 17, 1880.) Excess of ammonia is added to the solutions so as to redissolve the zinc hydroxide. Ferric and manganic oxide remain undissolved and can be separated by filtration. Steam is then blown into the filtrate, to distill off the excess of ammonia and to precipitate the zinc hydroxide. The latter is filtered off, dried and heated. The ammonia is recovered from the solutions of sulphate and chloride by adding lime and distilling.

C. SEMPER, Philadelphia: *Manufacture of aluminium sulphate.* (Eng. P. 2,997, July 7, 1881). Precipitates iron from the solution by adding potassium ferrocyanide and cupric sulphate.

BADISCHES, GESELLSCHAFT FÜR ZUCKERFABRIKATION, Waghäusel: *Improvements in the method of dry distillation of molasses swill for the purpose of obtaining swill char, ammonia, methylic alcohol and combustible gases.* (Germ. P. 15,702, Dec. 4, 1880). The evaporated swill is subjected to dry distillation with 5 to 15 per cent. of the dry substance of KOH obtained by causticizing with lime a portion of the char from a previous operation. The nitrogenous constituents of the swill are decomposed into simple ammoniacal compounds and a contamination of the potassium carbonate with lime avoided.

HARING, EHRENBURG & Co. and M. BASWITZ, Halle o/s: *Improvements in the dry distillation of molasses swill, elution lyes and osmose waters for the purpose of obtaining ammonia.* (Germ. P. 15,751, April 2, 1881). The liquid, concentrated to sp. gr. 1.35–1.39, is allowed to fall in drops into retorts heated to redness. The resulting gases are made to pass through a number of heated tubes located in the same furnace, in order to decompose the difficultly manageable tar, before they enter the receiver.

H. OPPERMANN, Bernburg: *Method of converting molasses swill into a dry powder with the acid of caustic lime.* (Germ. P. 16,033, Nov. 21, 1880).

## XVII. ON THE DETERMINATION OF REVERTED PHOSPHATES.\*

BY THOMAS S. GLADDING, A. M.

In an investigation on the subject of reverted phosphoric acid, the first question that arises is, What is meant by this term? The curious phenomenon of a decrease in the amount of soluble phosphate in a superphosphate, and its reversion into a form insoluble in water, but soluble in the organic salts of ammonia, in weak acids and other reagents, has been attributed to several different causes.

According to the most common explanation,† it is due to the gradual interaction between the soluble or monocalcic phosphate formed in the manufacture, and the still undecomposed tricalcic phosphate of the original rock, this reaction resulting in the formation of the intermediate compound dicalcic phosphate.

Again, it has been noticed that this reversion is especially large when the amounts of iron and alumina present are considerable, and in such cases it has been attributed‡ to the formation of phosphates of iron and alumina having the solvent properties specified above.

Again, from experiments in our own laboratory, it has been found that this reversion is very noticeable in acid phosphates, which, after treatment with sulphuric acid, have been mixed with natural phosphates containing large amounts of carbonate of lime. The reaction that takes place between the monocalcic phosphate and carbonate of lime probably results in the formation of either the dicalcic or tricalcic phosphate. This latter salt, though insoluble in water, is readily taken up, on account of its fine state of division, by the reagents used for the estimation of reverted phosphates.

From these different explanations of the character of reverted phosphate, it is evident that the term is not capable of a strict chemical definition. It may consist in any given case of one or of several chemical compounds. The best definition that can be given is the general one condensed into the name itself. Reverted phosphates are those which, originally insoluble, but having been made soluble, have reverted or returned to the insoluble condition. They must now, however, be looked upon as :

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\*Read before the American Chemical Society, March 17, 1882.

†Zeit. fur analyt. Chem. 10, Fresenius.

‡Zeit. fur analyt. Chem. 19, Z. Meyer.

(1.) Just over the border line that separates the soluble from the insoluble, lying very close to the soluble, and being brought back by a slight addition to the solvent power of water ; and, as

(2.) Widely separated and different in solubility from the insoluble form which they originally possessed. Like the flats lying just above the river's edge, they must be wholly covered by a slight rise in the water that affects the banks but little.

The object of the inquiry here described has been to establish the above distinctions, and to find a method of separation that shall in the actual analysis recognize them as nearly as possible. The course pursued has been that followed in similar inquiries :

I. An examination of the solubility of the different natural phosphates used in the manufacture of superphosphates.

II. An examination of the solubility of the different chemical compounds of which reverted phosphates have been found to consist ; and

III. An attempt based upon the results of these two inquiries to find the best method of separating these two classes.

In the examination of the natural phosphates, five different forms were taken as typical of all. These were :

1. *Apatite*, as type of the nearly pure tricalcic form in its most insoluble condition.

2. *South Carolina rock*, containing besides tricalcic phosphate, phosphates of iron and alumina, and carbonate of lime.

3. *Bone Ash*, the great organic source of phosphate.

4. *Navassa rock*, typical on account of its large percentage of iron and alumina ; and finally,

5. *Curacao phosphate*, as type of the island phosphates, notable for their peculiar mechanical condition and greater solubility in the organic salts of ammonia.

In examining the solubility of these natural phosphates, attention was first given to the effect of different relative proportions of phosphate and solvent. The standard method of Fresenius, Luck and Neubauer\* was followed, using 50 cc. of citrate of ammonia solution, of sp. gr. 1.09, and made as nearly neutral as possible ; time, 30 minutes, temp. 35° C. Two series of experiments were made. In the first, one gram of substance was taken ; in the second,  $\frac{1}{10}$  of a gram.

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\*Zeit. für analyt. Chem. 10.

	SERIES I.		SERIES II.	
	$P_2O_5$ dissolved.		$P_2O_5$ dissolved.	
Apatite	0.40 per cent.		1.54 per cent	
South Carolina rock	2.00	"	6.60	"
Bone Ash	2.60	"	8.30	"
Navassa	2.65	"	10.10	"
Curacao	4.40	"	16.00	"

Comparing these tables, we see at once the fact that has been commented upon very frequently of late by German chemists as regards superphosphates, and to which special attention has been called within a few months by a printed letter of Dr. S. W. Johnson, in the case of Curacao phosphate, namely, that the percentage of phosphate dissolved increases very largely with the increase of solvent, or, what is the same, the decrease of substance taken for analysis. These tables show that this is true not only of reverted phosphates as found by the former, nor of Curacao phosphate as shown by the latter, but also of all natural phosphates, including even the difficultly soluble apatite. This fact has a very important bearing upon the error involved in the determination of reverted phosphoric acid by this method, *i. e.*, an error as regards the strict separation of the reverted phosphates from the insoluble phosphates. Thus, supposing five superphosphates after the extraction of the soluble to contain no reverted phosphate, but to contain 10 per cent. of the above raw phosphates respectively, corresponding to about 3 per cent. of insoluble phosphoric acid, the citrate would attack these raw phosphates, and instead of the error being 0.04, 0.20, 0.26, 0.26 and 0.44 per cent. as would naturally be supposed from Series I, it would in reality be 0.15, 0.66, 0.83, 1.01 and 1.60 per cent. as shown by Series II. Thus, the superphosphates would be reported as containing these latter amounts of reverted phosphoric acid, when in fact they contained none whatever.

This same fact of increased solvent power obtained by increasing the amount of solvent salt was shown to be true also of oxalate of ammonia, by treating 2 grams of Navassa rock and Curacao phosphate with 1, 5, and 10 grams of the salt dissolved in 100 cc. of water, at a temperature of 100° C. for 30 minutes.

	SERIES III.		
	1 gram.	5 grams.	10 grams.
Navassa, $P_2O_5$ dis.,	2.20	8.75	12.15
Curacao, " "	2.50	12.30	17.60



The influence of increase of temperature upon the amount of rock dissolved was shown by treating one gram of the different phosphates with 100 cc. of water containing four grams of oxalate at 20°C. and at 100°C.

#### SERIES IV.

	Apatite.	So. Car. Rock	Bone Ash.	Navassa.	Curacao..
At 20°C. $P_2O_5$ dis.	0.86 pr ct.	2.25 pr ct.	2.72 pr ct.	2.85 pr ct.	5.51 pr ct.
" 100° " "	2.16 " "	5.80 " "	8.78 " "	9.40 " "	15.30 " "

In performing the above experiments at 100°C. a strong odor of evolved ammonia was observed, and it was also found that the filtrates were decidedly acid in reaction. The importance of this and its bearing upon the subject will be better seen further on.

The results thus far given show very clearly the cause of much of the confusion that has arisen over the determination of reverted phosphates. This lies in the great facility with which the citrate and oxalate solutions attack the insoluble phosphate, especially when the proportion of solvent is increased or when the temperature of digestion is raised. Many chemists have been led by these difficulties to seek a better method. The suggestion has frequently been made that a weak or dilute acid be employed as a substitute for the organic salts of ammonia. Thus Grupe and Tollens\* recommend the use of a  $\frac{1}{4}$  per cent. of citric acid solution. The solvent power of this acid of  $\frac{1}{4}$  per cent., also of oxalic acid of  $\frac{1}{4}$  per cent. and  $\frac{1}{10}$  per cent., also of hydrochloric acid of  $\frac{1}{10}$  normal

\*Berichte der deutsch, chem. Gesellschaft, 14, 754.

strength, upon the natural phosphates was examined. 100cc. of each of the above solvents were taken, the temperature of digestion was 40°C., time one hour. One gram was taken with each acid, and in addition also  $\frac{1}{10}$  of a gram with the citric acid of  $\frac{1}{4}$  per cent.

#### SERIES V

Acid employed—		Citric $\frac{1}{4}$ per ct.		Oxalic $\frac{1}{4}$ per ct.		Oxalic $\frac{1}{10}$ per ct.	HCl $\frac{1}{10}$ normal.
		1 gram.	$\frac{1}{10}$ gram.	1 gram.	1 gram.	1 gram.	1 gram.
Apatite,	$P_2O_5$ dis.	0.74 per ct.	2.70 per ct.	3.20 per ct.	1.08 per ct.	2.56 per ct.	
So. Car. Rock,	"	2.80 "	11.40 "	8.75 "	2.68 "	4.25 "	
Bone Ash,	"	3.80 "	11.80 "	9.23 "	4.15 "	3.85 "	
Navassa,	"	3.80 "	14.00 "	9.96 "	3.50 "	4.15 "	
Curacao,	"	4.20 "	19.80 "	9.12 "	4.00 "	3.35 "	

Supposing a fertilizer to contain 10 per cent. of insoluble rock, the citric acid would make the reverted phosphoric acid 0.27 per



cent., 1.14 per cent., 1.18 per cent., 1.40 per cent., 1.98 per cent. respectively in excess of the actual amount present. The excess thus introduced would be considerably greater than that caused by the solvent action of the citrate of ammonia solution, *vide* Series II., and the claim made for the citric acid solution by Grupe and Tollens that it dissolves nearly the same amount of phosphate from different quantities of substance taken for analysis shown to be wholly unfounded as regards its action on raw phosphates. (Compare columns 1 and 2 above.)

The action of the different solvents upon the dicalcic and tricalcic salts was next studied. These salts were obtained by precipitation, and on analysis found to agree so closely with the theoretical composition as to show the admixture of traces only of other forms. After thorough washing they were mixed with about an equal weight of calcium sulphate, in order to imitate the conditions under which they are found in a superphosphate. These mixtures were then thoroughly air-dried. One gram of each was now treated with the different solvents, already employed, for 30 minutes at a temperature of 40°C. The mixture of the dicalcic salt contained 19.20 per cent. of  $P_2O_5$ , of the tricalcic salt 13.00 per cent.

## SERIES VI.

Solvent.	$2CaO, P_2O_5$ .	$3CaO, P_2O_5$ .
100 cc. $\frac{1}{10}$ per cent. citric acid, $P_2O_5$ dis. =	$\left\{ \begin{array}{l} 10.60 \\ 9.31 \end{array} \right.$ per cent.	10.26 per cent.
100 cc. $\frac{1}{10}$ " oxalic acid, "	5.32 "	5.10 "
" $\frac{1}{100}$ normal HCl "	3.42 "	1.90 "
50 cc. cit. of ammonia sol. "	19.20 "	13.00 "
50 cc. $H_2O$ +1 gram oxal. ammo. "	$\left\{ \begin{array}{l} 19.00 \\ 19.05 \end{array} \right.$ "	11.50 "

It will be seen from this table that the citrate solution dissolves both forms perfectly ; that the citric acid, though fairly successful with the tricalcic salt, dissolves only one-half of the dicalcic salt ; that the other acids, notably the mineral acid HCl, fall far below the citric in this respect. Special attention is called to the vigor of the weak solution of oxalate of ammonia. Though only one gram is used, it dissolves all but a trace of the dicalcic salt, and falls but little short of dissolving all the tricalcic form. In the original article of Fresenius, Luck and Neubauer, the weak acids were discarded on account of their inability to take up the reverted phosphate. This conclusion is corroborated by these experiments, with the additional objection proved by Series V., namely, the greater extent to which they dissolve the insoluble phosphate. They found

also that oxalate of ammonia solution dissolved the dicalcic phosphate equally with the citrate salt, but abandoned its use on account of the vigor with which it attacked the raw phosphates. But they employed in their published experiments a very large amount of oxalate, six grams of the salt being taken. But it is here found that a much weaker solution of the oxalate is effective for dissolving the dicalcic salt. As to its solvent action upon the natural phosphates, a series of experiments proved that it was not as energetic in this respect as the citrate of ammonia solution, dissolving considerably less than the latter. On account of the many advantages possessed by an oxalate of ammonia solution over the citrate solution (1) as regards ease of preparing a perfectly uniform, neutral solution, (2) as to the greater facility of filtering and washing, and many others, many chemists urged and secured its adoption at the Cincinnati convention of agricultural chemists of 1881. But further experiments, to be described later, reluctantly compelled the conclusion that it cannot supersede the citrate of ammonia solution.

A series of experiments were now made upon a sample of acid phosphate, in order to compare the results obtained by the different methods, and to throw light, if possible, upon the causes of discrepancy. A sample made from Navassa rock was chosen on account of the large amount of reverted phosphate usually present in this class of fertilizers. The total phosphoric acid in the sample was 17.00 per cent., the soluble was 5.00 per cent., leaving 12.00 per cent. of reverted and insoluble. After thoroughly washing out the soluble, one gram was treated with 50 cc. of citrate of ammonia solution, and the directions of Fresenius carefully followed. Another gram was treated in exactly the same way, using however as a solvent 50 cc. of water containing in solution one gram of oxalate of ammonia. The following results were obtained:

#### SERIES VII.

	$P_2O_5$ dissolved.	$P_2O_5$ undissolved.
By citrate solution or "Washington method,"	6.10 per cent.	5.90 per cent.
By oxalate solution,	6.15     "	5.85     "

These two methods give results that are gratifying as regards their agreement. Subsequent experiments, however, showed conclusively that both were far from the truth.

Two grams of the same sample were now, after the extraction of

the soluble phosphate, treated according to the method adopted by the agricultural chemists at Cincinnati, known as the "Cincinnati method." The two grams of residue were washed from the filter paper into a breaker with 100 cc. of water, containing in solution two grams of oxalate of ammonia. (This is the same amount of oxalate in proportion to the quantity of substance taken as is used in the above determination.) 400 cc. of water were brought to boiling, and the breaker, closely covered, now introduced into the boiling water, the lamp being at the same time removed. The temperature of the liquid in the beaker rose to about 70° C. in a few minutes, and then slowly sank during an hour to about 40° C. The beaker was quickly rotated at intervals. Two grams were treated in exactly the same manner, using however, instead of the oxalate solution 100 cc. of the citrate solution.

#### SERIES VIII.

	$P_2O_5$ dissolved.	$P_2O_5$ undissolved.
By oxalate of ammonia,	8.55 per cent.	3.45 per cent.
“ citrate “	9.85 “	1.15 “

The point of interest connected with these results is the much larger amount of phosphoric acid dissolved by both the reagents as compared with that dissolved by them at the temperature of 35° C. continuous. This is especially the case with the citrate solution, the dissolved phosphoric acid rising from 6.10 per cent. to 9.85 per cent., an increase of 3.75 per cent. This increase can be explained in two ways only: (1) Either the insoluble rock present is dissolved to this increased extent, or (2) there is a form of reverted phosphate present that is not fully taken up by either of the citrate or oxalate solution at the lower temperature.

In order to ascertain if the former of these two explanations was the correct one, two separate portions of  $\frac{2}{3}$  of a gram each of Navassa rock, containing 0.120 gram  $P_2O_5$ , and corresponding to 6 per cent. of insoluble phosphoric acid when calculated on two grams as used in the above analysis (which is just the amount of apparently insoluble acid left in Series VII.), were rubbed to a paste in a mortar and treated each with 100 cc. of citrate of ammonia solution, the one at a temperature of 35° C. continuous for one hour, the other at a temperature falling from 70° C. to 40° C. during an hour, as in Series VIII. The following amounts of  $P_2O_5$  were dissolved, calculated as per cent. on 2 grams:

## SERIES IX.

No. 1.	Phos. acid dissolved,	1.16 per cent.
No. 2.	“ “ “	1.59 “

The increase in phosphoric acid dissolved by the use of the higher temperature is found to be only 0.43 per cent. It is seen at once that this small increase cannot explain the large increase of 3.75 per cent. in the case of the acid Navassa, made from the same rock.

The second of the two possible explanations, viz.: The presence of a form of reverted phosphate not fully dissolved at 35° C., must therefore be the true one. The experiments already made show that this form can be neither the dicalcic nor tricalcic phosphates, as the citrate solution is a perfect solvent for these two forms at a temperature of 35° C. The only form left is the reverted phosphates of iron and alumina. A quantity of the mixed normal phosphates of iron and alumina,  $\text{Fe}_2\text{O}_3$ ,  $\text{P}_2\text{O}_5$  and  $\text{Al}_2\text{O}_3$ ,  $\text{P}_2\text{O}_5$ , was now prepared by precipitation. A portion was mixed with a large quantity of calcium sulphate and dried to a powder at a temperature not exceeding 60° C. The remainder was dried at the same temperature without admixture. One gram of the first portion containing 4 per cent. of phosphoric acid was now rubbed to a paste in a mortar and then digested with 100 cc. of the oxalate and citrate solutions, first at 35° C. and then at 70° C. to 40° C. for one hour.

## SERIES X.

	$\text{P}_2\text{O}_5$ dis. at 35°C.	$\text{P}_2\text{O}_5$ dis. at 70° to 40°C.
Using oxalate of ammonia,	0.95 per cent.	3.20 per cent.
“ citrate “	1.60 “	4.00 “

One gram of the unmixed portion containing on analysis 35.62 per cent. of phosphoric acid was now treated exactly in the same way, with results as follows :

## SERIES XI.

	$\text{P}_2\text{O}_5$ dis. at 35°C.	$\text{P}_2\text{O}_5$ dis. at 70° to 40° C.
Using oxalate of ammonia	11.06 per cent.	26.50 per cent.
“ citrate “	13.14 “	35.62 “

These two series of experiments prove the very important fact that neither the citrate nor oxalate solution will dissolve more than a small portion of the reverted phosphates of iron and alumina at the temperature of 35° C., and that even at the higher temperature

of 70° C. to 40° C. the oxalate falls short of the perfect solvent power which must be required of it.

These results explain most clearly the great increase of phosphoric acid taken up in Series VIII. over that dissolved in Series VII. The acid Navassa under treatment contains a very large quantity of reverted phosphates of iron and alumina which are not dissolved at the lower temperature. They also explain why the citrate solution dissolves in 1.30 per cent. more than the oxalate, because of its more vigorous solvent action on these forms of reverted phosphates. One gram of these phosphates of iron and alumina, treated with 100 cc. of the citrate solution at a continuous temperature of 65°C. for 30 minutes, formed also a clear solution.

In order to ascertain the amount of solvent action of this method upon the different forms of raw phosphates,  $\frac{1}{2}$  of a gram of each, corresponding to about 3 to 4 per cent. phosphoric acid when calculated upon two grams, was treated in the same manner as the above, temperature 70°C. to 40°C., using 100 cc. citrate solution.

## SERIES XII.

	P <sub>2</sub> O <sub>5</sub> dis.
Apatite	0.22 per cent.
South Carolina rock	0.61 “
Bone Ash	....
Navassa rock	1.09 “
Curacao	2.07 “

Comparing this table with Series II., dividing the results there by by ten, it is evident that at the higher temperature, only a small increase in the amount of insoluble rock dissolved is observed.

In this connection special attention is called to one point. Notice has been called to the fact that at 100°C. the oxalate solution loses ammonia and becomes decidedly acid in reaction. A solution of neutral oxalate, immersed in boiling water for one hour in an open beaker, acquired an acid strength of  $\frac{1}{2}$  per cent. oxalic acid; 100 cc. of the citrate of ammonia solution treated for an hour in the same way acquired an acid strength of 1.2 per cent. citric acid. The energy with which these acid solutions attack the undecomposed rock present in a superphosphate is shown in Series V. It is on account of these facts that Fresenius uses a neutral solution of the citrate salt and adopts the low temperature of 35°C., and Luck\* in a second paper dwells with great emphasis upon this point in answer to the criticisms of a French chemist. But as the higher

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\*Zeitschrift fur anal. Chem. 14.

temperature is rendered indispensable on account of the presence of phosphates of iron and alumina in the great majority of superphosphates, this loss of ammonia and consequent acidity was prevented by digestion in a flask closed with a rubber cork.

The severest criticism upon the determination of reverted phosphates as an analytical process is the fact that when different quantities of substance are taken for analysis, different results are obtained. This most exceptional experience in chemical analysis, which is without a parallel in analytical determinations, requires a most careful examination, as this has done more than all else to cast discredit upon this analytical process. And yet it seems clear that this trouble can arise from only three sources :

(1.) Either the fertilizer under treatment has so large an amount of reverted phosphate present that when a large amount is taken for analysis the citrate is unable to take it all up, but is able to do so when a smaller amount is employed, or—

(2.) The citrate is an imperfect solvent for some form of reverted phosphate that may be present, either on account of the character of the latter, or on account of the method employed failing to dissolve it completely, the error showing more clearly as large quantities are taken for the analysis, or—

(3.) The discrepancy arises from the solvent action of the citrate upon the undecomposed rock present, a larger percentage of this being dissolved when smaller quantities are taken (*vide* Series I. and II.) The instances given by German experimenters, in the case of fertilizers containing 25 per cent. or more of phosphoric acid present as precipitated tricalcic phosphate, may be due mainly to the first of these causes. In such cases a smaller quantity must be taken, or it may be found that the higher temperature here recommended will so increase the solvent power of the citrate solution as to render this unnecessary. Whenever the phosphates of iron or alumina are present, it is evident from the experiments given that this discrepancy may be largely due to the second cause here assigned, namely, the method of the analysis, employing as it does the low temperature of 35° C. In order to ascertain whether the higher temperature of 70° C. to 40° C. will obviate the error from this second cause, 1, 2, 3, 4, and 5 grams of the above acid Navassa were treated each with 100 cc. of the citrate solution at this temperature, after the thorough washing out of the soluble phosphate.

## SERIES XIII.

			P <sub>2</sub> O <sub>5</sub> dissolved.	P <sub>2</sub> O <sub>5</sub> undissolved.
Employing 1 gram substance			10.06 per cent.	1.94 per cent.
“ 2	“		9.85 “	2.15 “
“ 3	“		9.65 “	2.35 “
“ 4	“		9.43 “	2.57 “
“ 5	“		9.02 “	2.98 “

The very slight and uniform decrease of reverted phosphoric acid, about 0.20 per cent. for each additional gram taken, up to the 5 grams when the increase is a trifle larger, would certainly seem to show that every source of discrepancy but the third mentioned above had been obviated. When it is observed that in the last experiment 0.451 gram of reverted phosphoric acid is dissolved by the 100 cc. of citrate of ammonia solution at the temperature of 70° C. to 40° C., it certainly seems that nothing further could be desired as regards vigor of action upon the reverted forms. No other solvent suggested will approach the citrate solution in this important respect, when employed at the temperature here adopted.

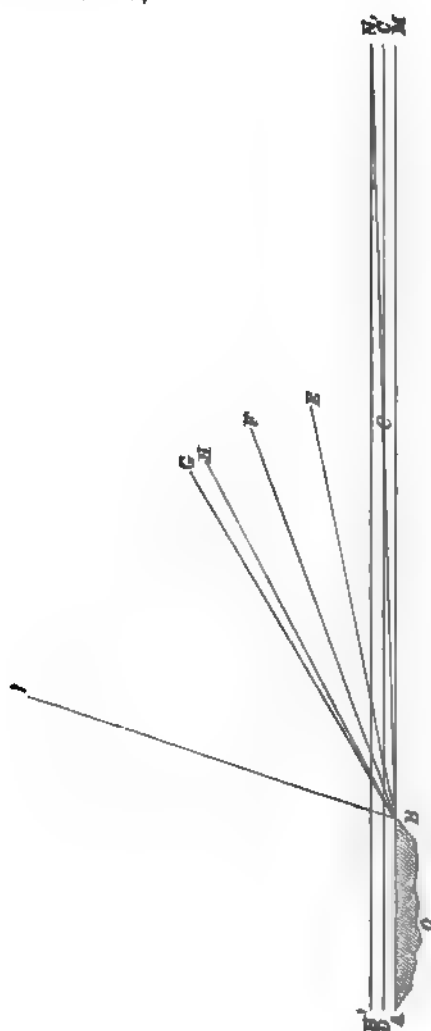
But as regards the third cause of discrepancy it is very different. This cause lies in the very nature of things and cannot be overcome. No reagent can be found that will dissolve the strictly reverted phosphates and not affect the undecomposed rock present. Nor is it desirable that any such reagent should be found, nor would it be just to employ it in case it were possible to find it. The whole idea upon which the determination of reverted phosphates rests is that the solvent action of the liquids of the soil is stronger than that of pure water, and is closely imitated by the solvent action of the organic salts of ammonia. Now there is a certain percentage of the raw phosphates that will always be taken up by this increased solvent power, and this percentage is practically and with justice considered equal in value to and reported as reverted phosphate.

The only difficulty that now presents itself is to so fix the amount of substance taken for analysis that the solvent action of the citrate solution upon the undecomposed rock shall dissolve such a percentage of this as shall when reported as reverted phosphate be a fair equivalent of its agricultural value. This conception here stated will be best illustrated by the accompanying diagram. 2 grams of the five forms of raw phosphates were treated with 100 cc. of citrate of ammonia solution at a falling temperature 70° C. to 40° C. for one hour, also two grams of the reverted phosphates

of iron and alumina containing 35.62 per cent. of phosphoric acid. The latter was dissolved to a clear solution.

SERIES XIV.

Apatite,	Phosphoric acid dissolved,	0.60 per cent.
South Carolina rock,	" "	2.62 "
Bone Ash,	" "	3.15 "
Navassa,	" "	3.75 "
Curacao,	" "	7.50 "
Reverted phos. of iron and alumina,	" "	35.62 "



In the diagram use is again made of the figure of speech previously employed. AOB is the river bed, representing the phosphates soluble in water, completely covered by the line AM, the height of which represents the solvent power of water. The line BC represents the flats or the reverted phosphates lying just above the water, and slightly rising, since the different forms possess different degrees of solubility. The line DC, DC' represents the solvent power of 100 c.c. citrate of ammonia solution at the temperature 70° C. to 40° C. The lines BE, BF, BH, BG and BI represent the banks rising at different angles on the different raw phosphates, Curacao, Navassa, Bone Ash, South Carolina rock, and Apatite here examined. These lines are all two inches in length, to represent the two



grams of substance taken. All of BC falls below DC or becomes soluble in the citrate solution,  $\frac{1}{4}$  of BE,  $\frac{1}{8}$  of BF,  $\frac{1}{11}$  of BH,  $\frac{1}{16}$  of BG, and  $\frac{1}{16}$  of BI, calculating from 30, 30, 36, 27, and 42 percentages of phosphoric acid present in the five samples.

Now suppose one gram of the above substances be taken to 100 cc. of citrate solution. This proportion is the same as 200 cc. citrate solution to the 2 grams of substance taken in the diagram. The line DC will be raised to H'N, twice as far above the surface of the water. Twice as large an amount of reverted phosphates will be taken up, viz. BN, and at the same time nearly twice as large an amount of the raw phosphates as shown in the figure. Thus repeating Series XIV., using however one gram of substance, there were dissolved as follows:

## SERIES XV.

Apatite,	Phosphoric acid dissolved,	1.06 per cent.
South Carolina rock,	" "	
Bone Ash,	" "	5.20 "
Navassa,	" "	6.20 "
Curacao,	" "	11.50 "

Attention is called to the nature of Curacao phosphate as brought out in this connection. Since by successive treatments with citrate of ammonia solution, or by employing a very small quantity for analysis, as  $\frac{1}{16}$  of a gram, nearly all of this can be brought into solution, many chemists regard its phosphoric acid as nearly all in the available form, and an analysis made by one chemist on the identical sample used in this work reports the presence of 18 per cent. of reverted phosphoric acid. But the whole course of experiments given in this paper shows conclusively that it differs from the other raw phosphates only in degree of solubility, and is very far removed as regards this property from the true reverted forms, being, as shown in the diagram, based upon Series XIV., only  $\frac{1}{4}$  as soluble at the best as the latter, and only about twice as soluble as Navassa rock and bone ash.

It seems almost certain from the agricultural experiments described in the last edition of Johnson's "Agricultural Chemistry," on the use of finely ground phosphates, that all the phosphoric acid in such phosphates becomes ultimately soluble in the soil. The division of phosphates therefore into the three forms, soluble, reverted, and insoluble, will have reference only to the time required to render them available for plant use. Supposing, in the

above diagram, that the solvent action of 100 c.c. of the citrate solution represents the solvent action of the liquids of the soil for a certain period of time, as *e.g.* one season, upon the forms of phosphate there represented, it is evident that all the soluble and reverted would be available the first season, that raw Curacao would all be rendered available in something over five seasons, the Navassa in eight seasons, and so on. The divisions, therefore, of soluble, reverted, and insoluble become indispensable as means of fixing commercial and agricultural values.

In conclusion, emphasis is laid upon the following points as brought out in this inquiry.

I. While the line of division between the reverted forms and the original insoluble forms of phosphoric acid may be sharply drawn in the case of an extremely insoluble phosphate, like apatite, in the case of the more easily soluble natural phosphates the two great classes overlap, one the other. Whatever solvent therefore is used for determining reverted phosphates the same will also dissolve a small percentage of the undecomposed phosphate that may be present; this percentage varying considerably according to the character of the insoluble phosphate. This percentage, representing as it does the comparative solubility which such insoluble phosphate possesses to the true reverted phosphates present, is justly considered as equivalent to an equal amount of the true reverted forms, and reported as such.

II. A slightly ammoniacal citrate of ammonia solution, alone of all the solvents that have been proposed, is a perfect solvent for all the forms of reverted phosphate, while at the same time not unduly dissolving the raw or insoluble phosphates present. Such a solution dissolves but a trace of the most difficultly soluble form of phosphate known, viz. apatite, and dissolves the other natural phosphates to a greater and greater extent as they approach in character the true reverted forms.

III. Neither the "Washington method" (method of Fresenius) nor the "Cincinnati method" can be relied upon to take up all the reverted phosphates that may be present.

IV. A modification of the method of Fresenius, consisting of the use of a higher temperature for the digestion, will meet all the requirements of the case, provided that the greatest care is taken to guard against acidity of the solvent and consequent excessive solution of the insoluble phosphates present. Whether the falling temperature of 70° C. to 40° C. as here explained, or a continuous tem-

perature of 65° C. for 30 minutes, be used, there will be but slight if any difference in the results, provided that the solution of citrate be slightly ammoniacal, and the digestion be made in a closed flask. To the use of *acid* solutions of citrate, either acid at the beginning or the digestion or becoming acid during its continuance, are probably due more than to any other cause, the discrepancies in this analytical process.

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## XVIII. THE CONTAMINATION OF THE NEW YORK WATER SUPPLY.

BY DR ALBERT R. LEEDS

In this journal (vol. III, 98,) I have given under the title "Relative purity of city waters in the United States," the analyses of the drinking waters of many of our large cities, as performed by the same methods, and as made upon samples collected at or near the same date. As a necessary deduction from these results, the drinking waters arranged themselves in a series, in which the Brooklyn water occupied the place of honor as the purest, the Boston water the lowest place, being the most foul. The New York water, No. VI, fell in the middle of the series. The water supplies (judged by the samples analyzed) of Brooklyn, Rochester, Philadelphia, Baltimore and Washington, were pronounced satisfactory. Those of New York, Newark, Jersey City, Hoboken, Cincinnati, Oswego, Wilmington (Del.) and Boston, were stated to be contaminated. There was no question as to the contamination of the Newark (Passaic) water (No. 7), because its impurities were established by a multitude of analyses. And with regard to the Croton water the evidence of contamination was found in the facts:

1st. That its various impurities exceeded the limits which are allowable in a pure drinking water. 2nd. That its composition approached very closely to that of the Passaic, of whose contamination there could be no question; 3rd. That the water shed of the Croton contained sufficient sources of pollution to account for the figures obtained by analysis.

The results above summarized have been accepted without cavil, except in the case of the New York water supply. The pollution of the Boston water was so great that I could not credit my results without making a personal inspection of the sources of the Boston supply. This I did at a later season, and found not only were the first results more than confirmed but that the sources of pollution

were unmistakable. They were in the first place, the impurities thrown in by manufacturing communities ; and secondly, the enormous expansion of certain organic growths at favorable seasons of the year. The results relating to the water supply of Wilmington, Del., which were at first objected to by the uninformed local press of that city, were later on acknowledged by the health and city authorities to be warranted by the actual facts, and I am at present engaged on behalf of this city to investigate the precise nature and extent of the pollution.

Dr. E. Waller in this journal (Vol. IV, p. 15), has objected to the New York water supply being characterized as contaminated, on the ground that his results, for a term of years, differ greatly from my own and are of a more favorable character. Also, for the reason that the Croton water shed cannot originate any sources of contamination ; and finally, that the health of New York is not, and has never been, such as to indicate the presence of any contamination in the water supply.

In reply, I would state :

1st. That the analyses of the New York water, on which Dr. Waller bases his opinion, have been incomplete, and data have been lost sight of, which are of the greatest value in forming a judgment as to the fact of contamination.

2nd. That certain of his methods are erroneous, and of necessity give a more favorable impression of the water than its true character warrants.

3rd. The analysis of the Croton water made June 23rd, 1881, does not stand alone, but is confirmatory of other previous analyses which led me to the conclusion that the Croton water was contaminated. It was not made, as Dr. Waller intimates, upon water turbid with sediment, but upon clear water drawn from the continuously flowing faucet in the Barclay street ferryhouse. I took it from this source as affording a very fair average sample of Croton water and I have no reason for supposing that it was otherwise.

4th. The condition of the Croton water shed is such as to account for the figures given in my analysis. The table of population given by Dr. Waller for the water sheds, does not explain the facts which exist ; for Rochester with but *half* the density of population on its Hemlock Lake water shed, that exists upon the Croton, has a far superior water supply, while Brooklyn on the other hand, with *double* the population, has a better supply than either.

5th. There are no data given, or so far as I know ever have been

given, upon which conclusions can be based connecting the vital statistics of New York with the character of its water supply. The face of Dr. Waller's assertion is not to be measured by its positiveness, but upon the appositeness and weight of the data upon which it is based, and of such data he has given us none.

It is extremely difficult, unfortunately, to connect the various diseases of a great community with the more or less deteriorated condition of its water supply. Unfortunately because if the connection were one easily proved, both chemist and medical practitioner could arrive with ease and certainty at results which are now open to more or less criticism. Whilst reiterating in various communications the statement that the water supply of Newark and Jersey City is contaminated, and in seasons of excessive drought like the summers of 1881 badly so, I have repeatedly challenged the physicians of these towns, to connect by irrefragible demonstration, the impurity of the water with any case of disease, and they have professed their inability so to do. I mention this, more particularly because the chemist is often accused by the physician of reporting a long series of decimals, without satisfactorily showing that any of the substances reported are specific causes of disease. Here, on the other hand, is a case where for years the water has been reported, on the strength of the oxidizable and oxidized nitrogenous matters which it contains, to be contaminated, and yet no physician has proven, or attempted to prove, that any particular case of disease has been due to drinking it. In the population of 50,000 people inhabiting the town of Paterson, which pours all its sewage into the Passaic at a point 16 miles above the intakes of the pumping stations of Newark and Jersey City, and in the city of Passaic with its considerable population likewise sewerage into the Passaic river at a point only half this distance, there must be many cases of typhoid fever, yet no one has ever shown that a single case of typhoid fever has been originated in Newark and Jersey City by drinking the Passaic water. From what source then does Dr. Waller derive the statistics upon which he infers the purity of the Croton water from the death-rate of the New York population?

To return, however, to the more serious sources of error in the methods made use of by Dr. Waller.

1st. With reference to the incompleteness of his analyses. He has charged me with indefiniteness in the use of the terms "nitrites" and "nitrates." I fail entirely to see any indefiniteness. I used these terms because they were the proper generic names of the sub-

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1st. With reference to the incompleteness of his analyses. He has charged me with indefiniteness in the use of the terms "nitrites" and "nitrates." I fail entirely to see any indefiniteness. I used these terms because they were the proper generic names of the sub-



substances I determined. Nitrites and nitrates were the bodies meant, and the bodies whose determination *as such*, I regarded as of importance. The results were calculated to nitrites ( $\text{HNO}_2$ ) and nitrates ( $\text{HNO}_3$ ), and not to the "nitrogen in nitrites or nitrates," because I had no interest in knowing the amount of the nitrogen, could not have made use of it in my calculations, and think its estimation would have been absurd. If the organic carbon and nitrogen had been determined instead of the free and albuminoid ammonia, then indeed I should have calculated out the nitrogen in the nitrates, in order to compare this amount with the organic nitrogen, and thus to find the correct ratio between the organic carbon and nitrogen in the organic compounds present.

There is no evidence that Dr. Waller had estimated the nitrates until the summer of 1881, nor does he appear to have remarked the fact that the Croton water at times gives the reaction for *nitrous acid*. The latter I regard as a most important fact (although too frequently overlooked), because it is very rare that a decided reaction for nitrites is obtained in drinking-waters, and only when the oxidizable nitrogenous compounds are caught in the very transient stage of nitrites, and before their oxidation to the final condition of nitrates. If in *river-waters* the reverse transformation is effected, and any nitrites undergo reduction to nitrites, I have not yet in my own experience encountered a case in which there was any reason for supposing that such a reverse transformation has been effected.

An unusual opportunity for noting this oxidation of albuminoids into nitrites and finally into nitrates, is afforded when the pure mountain-stream waters of the Passaic, after leaping the Great Falls, come straightway into contact with the sewage of the population of 500,000 inhabitants of Paterson. The albuminoid ammonia increased *per saltum* (Sept. 6th, 1881) from 0.014 parts per 100,000, to 0.225 parts. It then fell away in the course of 2 miles flow to 0.133 parts, and at the end of 7 miles flow had fallen to 0.0215 parts per 100,000. The nitrates at the corresponding points were 0.185 parts; 2.183; 0.722; 1.388 parts. The nitrites at the same points were none; a considerable amount (the quantitative determination of which was lost); a trace; none.

It is seldom, however, that the reaction for nitrites is obtained in the Passaic water after it has flowed 16 miles, and has been delivered by the pumping-stations to the consumers in Newark and Jersey City. The oxidation of the albuminoids during its long flow is so complete, that while the amount of nitrates is always



high, the amount of nitrites is usually quite inappreciable. And the same is generally true of river-waters. In the 23 samples of water, the composition of which is given in the table upon the "Comparative Purity of City Waters in the United States," no reaction for nitrites happened to be obtained in the case of any one of them. And yet the Boston water required the enormous amount of 1.77 parts to the 100,000 of oxygen to effect the oxidation of its organic matters, and contained 1.24 parts of nitrates. Sometimes the Passaic water does afford the nitrous reaction, but in these cases I have attributed it to the non-oxidized sewage of Newark which has only to flow upward for a couple of miles before reaching the intake of the pumping-stations, rather than to the sewage of Paterson, which has to flow downward 16 miles before reaching the same points, and is usually completely oxidized in its long exposure.

The fact that at times I have obtained from the Croton the nitrous reaction, in one instance a reaction strong enough to admit of quantitative estimation, has had due weight in forming my estimation of its purity.

2nd. The method which Dr. Waller employed of determining the amount of oxygen required to oxidize the organic matter is erroneous, and of necessity gives a more favorable impression of the water than its true character deserves. In making this statement, I am aware of what has been written in favor of this method, but see no reason for regarding it otherwise than erroneous. If it is desired to oxidize the organic matter in drinking waters by potassium permanganate, so as to destroy the possibly noxious substances, the water to which the potassium permanganate has been added is boiled before it is thought entirely safe to drink. If the potassium permanganate is to be standardized by an organic substance, as it is in this instance by oxalic acid, the standardization is performed by heating the solution of oxalic acid or of permanganate. But in making the determination of the organic matter in the water itself, this method proposes it should be done at a variable common temperature. Of course, only a small amount of the organic matter in the water is thus oxidized, and although it is claimed that this part is the really dangerous putrescible portion, the fact still remains that only a part of the organic matter is attacked by the permanganate in the cold, and the method is thus far erroneous. Still more, the method lacks the essentials of an accurate volumetric estimation, and does not admit of arriving at a certain end reaction. I tried the method, and after satisfying myself of its essential

inaccuracy, adopted what appeared to me the most satisfactory and definite process, that of titration at the boiling point.

Dr. Waller's objections to this method are fanciful rather than real. The impurities in the permanganate and sulphuric acid, though actually present, are too small to admit of any appreciable influence on the results. The objection that the chlorides in the water would affect the results has greater weight, but in practice these errors, like those produced in Tidy's modification of the permanganate process by the presence of chlorides, are too small to be considered. I am fortunately able to bring an experiment, performed on polluted natural waters themselves, to confirm this statement. The Passaic water (Sept. 6th, 1881), at the intake of the Jersey City Reservoir, contained in 100,000 parts:

	LOW TIDE.			HIGH TIDE.		
	E. Bank.	Middle.	W. Bank.	E. Bank.	Middle.	W. Bank.
Chlorine,	5.73	2.10	3.40	55.88	44.75	59.75
Oxygen } required }	0.55	0.44	0.44	0.47	0.60	0.76

The differences had been caused by the backing up of the river waters, on meeting the tidal wave from the sea.

On the same day, and upon the same two tides, the differences for the corresponding points at the Newark Reservoir, three-quarters of a mile further up stream, were :

	LOW TIDE.			HIGH TIDE.		
	E. Bank.	Middle.	W. Bank.	E. Bank.	Middle.	W. Bank.
Chlorine,	2.185	1.17	2.505	21.13	25.00	14.50
Oxy. required,	0.62	0.56	0.43	1.80	2.82	2.82

But it may be objected to these results, that the oxydizable organic matters in the various samples varied greatly, so that no inference could be drawn with regard to the influence of the chlorine itself.

This objection does not apply to the water taken at intervals of about a mile down the stream, upon the same day (Sept. 6th, '81).

Beginning at a point below the Tail Race at Passaic Mills, we found for samples :

	No.	75	76	77	78	79	80	81	82	83	84
Chlorine,		0.585	0.60	0.94	0.57	1.57	1.005	0.90	0.56	1.10	2.505
Oxy. required		0.53	0.50	0.49	0.52	0.51	0.59	0.47	0.47	0.47	0.43

It may be objected, however, that all these results merely raise a strong presumption in favor of the proposition that the small

amounts of salt in river water do not appreciably raise the figures obtained by permanganate at the boiling point. They do not prove it ; for proof a suitable experiment is demanded.

In the first place, it was necessary to determine the coefficient of change, when potassium permanganate is boiled with *distilled* water. Both in the cold and on heating, potassium permanganate changes in acidulated solution with distilled water, and the amount of this change has to be determined in both cases, and a corresponding deduction has to be made from the amount obtained with an equal volume of the natural water under examination, I thus obtained :

100 cc. distilled water, Miller's method (3 hr.) 0.9 cc. permanganate.

100 cc. distilled water, Kubel's method, — — 0.9 cc. permanganate.

Another test by Kubel's method on a different sample of distilled water, and with different permanganate solution, gave 1.15 cc. permanganate.

In other words, in order to make use of Miller's method with any expectation of arriving at accurate results, it is necessary in each case to apply a correction due to the alteration of the permanganate alone. This and many other precautions C. M. Tidy (Jour. London Chem. Soc., xxxv., p. 46) has taken, in order if possible to bring Miller's process into a form capable of yielding regular and accurate results. But I do not find in Dr. Waller's account of Miller's method, as he employed it, that any such precautions were taken. The influence of these corrections may be perceived by considering the great changes thereby produced in Dr. Waller's results. For instance, for the first five months of the year 1879, he obtained 0.072 parts of oxygen per 100,000, as the amount required to oxydize the organic matter. This would be equal to 0.72 cc. permanganate solution used. If, as is possible, he had obtained 0.9 cc. as the correction to be applied by the use of the permanganate solution with *distilled water alone*, there would have been left a minus quantity, or 0.018 parts of oxygen, as the amount required to oxydize the organic matters in the Croton water.

Dr. Tidy has endeavored to find the extent to which the chlorides affect the accuracy of the modified Miller's method, and has found that their influence, even when present in very large quantities, practically *nil*. A similar remark applies to the effect of chlorides on the results afforded by Kubel's method. For instance, in an experiment in which 100 cc. of *distilled water* decolorized on boiling 1.15 cc. permanganate, it decolorized only 1.3 cc. permanganate

after sodium chloride had been added in amount corresponding to 400 parts in 100,000. This amount vastly exceeds any quantity likely to exist in river or well waters.

In practice, however, I think it is better, whenever the amount of sodium chloride is large, to add the percentage indicated by the chlorine determination to the distilled water used in making the blank experiment. If the results thus obtained differ from those obtained by the use of the 100 cc. distilled water alone, they can be deducted, instead of the latter, from the number of cubic centimeters of permanganate obtained with the natural water.

3d, and finally. The analysis of the Croton water made June 3d, 1881, does not stand alone, but is confirmatory of other previous analyses, which led me to the conclusion that the Croton water was contaminated.

The best proof of this statement is to be found in an analysis made only three days before the one intended for the general comparison with the drinking water of all the other large cities, or on June 20th, 1881. It was made upon unfiltered but perfectly clear water, drawn from the same place, the constantly running faucet in the Barclay street ferry-house. It shows by its close agreement with the results obtained on the sample taken June 23d that neither analyses gave high figures by accident, but both are to be regarded as mutually confirmatory results of the actual average condition of Croton water at that time.

	CROTON WATER. PARTS PER 100,000.	
	June 20th, 1881.	June 23d, 1881.
Free ammonia.....	0.004	0.0027
Albuminoid ammonia.....	0.027	0.0270
Oxygen required.....	0.73	0.810
Nitrites.....	None.	None.
Nitrates.....	0.6105	0.8325
Chlorine.....	0.26	0.35
Total hardness.....	3.00	3.30
Permanent hardness.....	2.60	—
Temporary hardness.....	0.40	—
Total solids.....	9.00	11.80
Mineral matters.....	2.50	5.00
Organic and volatile matters	6.50	6.80

## XIX.—ON THE ESTIMATION OF PHOSPHORIC ACID AS MAGNESIC PYROPHOSPHATE.

BY THOMAS S. GLADDING.

In a valuable paper on the determination of phosphoric acid as magnesian pyrophosphate (*Amer. Chem. Journal*, vol. 1, 391) Gooch has demonstrated that this method, far from being as accurate as is generally supposed, is peculiarly liable to sources of error, which cause a considerable overestimation of the phosphoric acid actually present.

The great importance of this analytical determination from a commercial point of view, gives enhanced interest to any inquiry as to its accurate execution. In view of the vast capital at present engaged in the conversion of phosphatic materials into artificial fertilizers, and in view of the fact that chemists frequently differ so widely in this important determination, the necessity, both for the interests of commerce and the credit of science, of an accurate and rigid method can be clearly understood. In the case of large and valuable cargoes sold upon chemical analysis, the slight error of even  $\frac{1}{8}$  of a per cent. in the estimation of phosphoric acid causes a difference in value ranging from twenty-five up to two hundred dollars.

I have been led by these considerations to a careful examination of the modes of procedure at present recommended by the best authorities and which are in common use among chemists.

In the latest American edition of *Fresenius' Quant. Analysis* the directions are, to add to the neutral or moderately ammoniacal solution of the phosphate, magnesia mixture in slight excess, and after letting stand for some time,  $\frac{1}{2}$  its volume of strong ammonia solution. In the case of a previous precipitation as phospho-molybdate of ammonia, this precipitate is dissolved in weak ammonia and the neutral or slightly ammoniacal solution, treated as above.

The method recommended by the convention of chemists of the different experimental stations of Germany (held in Dec. 1881) is translated as follows: (*Zeit. fur. Anal. Chem.* 21, p. 290) "The beaker glass is now placed under the funnel, the filter pierced with a platinum wire, the precipitate washed into the beaker glass with  $2\frac{1}{2}$  per cent. ammonia liquor, and dissolved by stirring with a glass rod, and then enough of the  $2\frac{1}{2}$  per cent. ammonia liquor added, to bring the volume of the liquid up to about 75 cc. To 0.1 gram phosphoric acid, 10 cc. magnesia mixture are now dropped in ac-

accompanied by a constant stirring up." In a foot note is added "a gradual addition of the magnesia mixture is advisable under all circumstances even when the ammoniacal solution of the molybdate precipitate is previously nearly neutralized by addition of hydrochloric acid." This mode of procedure differs from that given in "Fresenius" principally in the one point of *insisting on a gradual* addition of the magnesia mixture. In both methods a strongly ammoniacal magnesia mixture is employed and this is added to a more or less strongly ammoniacal solution of the phosphate.

On the other hand the method giving the best results in the hands of Gooch, consisted in the addition of a *neutral* magnesia mixture to a *neutral* solution of the phosphate or still better, especially in the case of a previous precipitation as molybdic salt, in a double precipitation of the ammonia-magnesia-phosphate.

In testing the above methods great care was exercised in the preparation of the standard solutions. These were made by the use of chemically pure microcosmic salt. Upon careful ignition of this salt in a covered platinum capsule 5.1415 grams gave 2.5110 grams of phosphate of soda as residue or 34.00 per cent. of phosphoric acid against 33.98 per cent. required by theory. With this salt, two solutions were prepared, the one containing 4.121 grams of the salt in a liter or 0.035 grams  $P^3 O^5$  in every 25 cc., the other 20.605 grams to a liter or 0.175 grams of  $P_2 O^5$  in every 25 cc. 25 cc. were measured off with great accuracy by means of a pipette holding exactly 25 cc. by careful weight test, when filled to the mark. This was then carefully rinsed out. The small neck of the pipette, makes it easy to measure a liquid to within  $\frac{1}{100}$  cc. by this method. In addition to the experiments made with these solutions, others were made by direct weighings of the microcosmic salt.

#### METHOD I.

To the solution of the phosphate, diluted to 50 cc. and made slightly *alkaline* by the addition of  $\frac{1}{2}$  cc. of ammonia liquor, the regular ammoniacal magnesium chloride mixture was added, 4 cc. to every 25 cc. of solution I. and 18 cc. to every 25 cc. of solution II. After letting stand for 15 minutes, 25 cc. of strong ammonia solution were added, and the liquid filtered off after about 30 minutes. The washing was performed with strong ammonia solution and was continued until 10 drops of the washings gave no precipitate with acid of silver nitrate when acidified. In these and all subsequent experiments the filter papers were each weighed, that

an accurate weight allowance might be made for filter ash, and the precipitate was strongly ignited over a blast lamp for several minutes, until white. Two series of experiments were made, the one without a previous precipitation as phospho-molybdate of ammonia, the other with such previous precipitation.

A. Without previous precip. as molybdate salt.

Amount taken.	Mg <sub>3</sub> P <sub>2</sub> O <sub>7</sub> found.	P <sub>2</sub> O <sub>5</sub> found.	P <sub>2</sub> O <sub>5</sub> found.	Error.
1 25 cc. sol. I.	0.0553	0.0350	0.03575	2.14 p. c.
2 "	0.0557	"	0.03563	1.20 "
3 "	0.0559	"	0.03575	2.14 "
4 "	0.0557	"	0.03563	1.80 "
5 25 cc. sol. II.	0.2770	0.1750	0.17718	1.25 "
6 "	0.2763	"	0.17673	0.99 "
7 "	0.2760	"	0.17654	0.88 "
8 "	0.2762	"	0.17666	1.95 "

Average error 1.50 per cent.

B. With previous precip. as molyb. salt.

Amount taken.	Mg <sub>3</sub> P <sub>2</sub> O <sub>7</sub> found.	P <sub>2</sub> O <sub>5</sub> required.	P <sub>2</sub> O <sub>5</sub> found.	Error.
1 25 cc. sol. I.	0.0559	0.0350	0.03575	2.14 p. c.
2 "	0.0559	"	0.03575	2.14 "
3 "	0.0558	"	0.03569	1.97 "
4 "	0.0556	"	0.03555	1.57 "
5 25 cc. sol. II.	0.2768	0.1750	0.17705	1.17 "
6 "	0.2780	"	0.17782	1.61 "
7 "	0.2774	"	0.17744	1.40 "
8 "	0.2776	"	0.17756	1.46 "

Average error 1.68 per cent.

METHOD II.

The same course was followed as in method I, excepting that the solution of phosphate was exactly NEUTRALIZED before the addition of the magnesia mixture.

A. Without previous precip. as molyb. salt.

Amount taken.	Mg <sub>3</sub> P <sub>2</sub> O <sub>7</sub> found.	P <sub>2</sub> O <sub>5</sub> required.	P <sub>2</sub> O <sub>5</sub> found.	Error.
1 25 cc. sol. I.	0.05570	0.0350	0.03563	1.80 p. c.
2 "	0.05570	0.0350	0.03563	1.80 "
3 25 cc. sol. II.	0.2758	0.1750	0.17641	0.81 "
4 "	0.2757	0.1750	0.17635	0.77 "
5 "	0.2753	0.1750	0.17609	0.62 "

Average error 1.16 per cent.

## B. With previous precip. as molyb. salt.

Amount taken.	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> found.	P <sub>2</sub> O <sub>5</sub> required.	P <sub>2</sub> O <sub>5</sub> found.	Error.
1 25 cc. sol. I.	0.0556	0.0350	0.03555	1.57 p. c.
2       "       "	0.0556	0.0350	0.03555	1.57 "
3 25 cc. sol. II.	0.2770	0.1750	0.17718	1.25 "
4       "       "	0.2772	0.1750	0.17731	1.32 "

Average error 1.43 per cent.

## METHOD III.

To the solution of phosphate, 75 cc. in volume and quite strongly ammoniacal, the magnesia mixture was added at the rate of one drop a second, the contents of the beaker being kept in constant rotation. The magnesia mixture was added from a burette, the rapidity of flow being regulated by a glass cock, or still better from an ordinary Mohr burette, the flow being regulated by a screw-clamp. The beaker was slightly inclined, so as to receive the drops on its side. The magnesia mixture by this arrangement flowed down the side of the beaker in a thin stream and an exceedingly gradual admixture with the phosphate solution was thereby secured. After the addition of the magnesia mixture, 25 cc. of strong ammonia were added and the whole allowed to stand for two or three hours. Very strong ammonia water was used in the washing as in several cases a tendency to run through the filter paper was observed, when a weaker ammonia solution happened to be employed. One part conc. ammonia water to three parts water was employed.

## A. Without previous precip. as molybdate salt.

Am't. taken.	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> found,	P <sub>2</sub> O <sub>5</sub> required,	P <sub>2</sub> O <sub>5</sub> found,	Error.
1. 25 cc. sol. I.	0.0547 g.	0.0350 g.	0.03499 g.	0.00 p.c.
2.       "       "	0.0547	0.0350	0.03499	0.00 p.c.
3. 25 cc. sol. II.	0.2736	0.1750	0.17500	0.00 p.c.
4.       "       "	0.2735	0.1750	0.17494	0.03 p.c.
5.       "       "	0.2738	0.1750	0.17513	0.07 p.c.
6. 0.5231 g. micro. salt	0.2775	0.17775	0.17750	—0.14 p.c.
7. 0.5120	0.2717	0.17397	0.17379	—0.10 p.c.

Average error. 0.05 p. c.



## B. With previous precip. as molybdate salt.

Am't. taken.	Mg : P <sub>2</sub> O <sub>7</sub> found,	P <sub>2</sub> O <sub>5</sub> required,	P <sub>2</sub> O <sub>5</sub> found,	Error.
1. 25 cc. sol. I.	0.0548 g.	0.0350 g.	0.03505	0.14 p.c.
2. "	0.0548	0.0350	0.03505	0.14 p.c.
3. 25 cc. sol. II.	0.2736	0.1750	0.17500	0.00 p.c.
4. "	0.2734	0.1750	0.17488	—0.07 p.c.
5. "	0.2732	0.1750	0.17475	—0.14 p.c.
6. "	0.2733	0.1750	0.17481	—0.11 p.c.
Average error. 0.10 c.				

## METHOD IV.

To the solution containing the phosphate, either neutral or ammoniacal, the magnesia mixture was added, and then 25 cc. of conc. ammonia water. After letting stand for thirty minutes, the liquid was filtered off and the precipitate thoroughly washed on the paper. The precipitate was then dissolved on the paper with dilute HCl, and washed into the original beaker. 25 cc. of conc. ammonia solution were now added to the filtrate, no care being taken to add it gradually. After the flocculent precipitate had settled, 5 cc. magnesia mixture were added and the whole allowed to stand several hours. The second precipitation was found to be incomplete without the addition of this extra magnesia mixture.

## A. Without previous precip. as molybdate salt.

Am't. taken.	Mg : P <sub>2</sub> O <sub>7</sub> found,	P <sub>2</sub> O <sub>5</sub> required,	P <sub>2</sub> O <sub>5</sub> found,	Error.
1. 25 cc. sol. II.	0.2736 g.	0.1750 g.	0.1750 g.	0.00 p.c.
2. "	0.2735	0.1750	0.17494	0.03 p.c.
3. "	0.2736	0.1750	0.17500	0.00 p.c.
4. 0.5095 g. micro salt.	0.2712	0.17313	0.17336	0.13 p.c.
5. 0.5703 "	0.3030	0.19379	0.19380	0.00 p.c.
6. 1.0108 "	0.5372	0.34347	0.34361	0.04 p.c.
Average error. 0.03 p. c.				

## B. With previous precip. as molybdate salt.

Am't. taken.	Mg : P <sub>2</sub> O <sub>7</sub> found,	P <sub>2</sub> O <sub>5</sub> required,	P <sub>2</sub> O <sub>5</sub> found,	Error.
1. 25 cc. sol. II.	0.2738 g.	0.1750 g.	0.17512 g.	0.00 p.c.
2. "	0.2736 g.	0.1750 g.	0.1750 g.	0.07 p.c.
Average error. 0.035 p. c.				

Still another method was investigated as follows: To the *neutral* solution of phosphate was added a *neutral* magnesia mixture in slight excess. No precipitate appeared except in the case of very conc. solutions. An extremely weak ammonia water was then added, drop by drop, to the liquid, to which was imparted at the same time a constant rotary motion. An exceedingly granular, pure precipitate was thus produced. The results obtained were perfectly accurate, but as the precipitate was very light and apt to fly, when removed from the paper into the capsule, for ignition, and as the method involved the extra labor over Method III, of first neutralizing the solution, it was not carried out in full.

A series of determinations by these several methods, on different fertilizers received in the course of business, was also carried out. The average error is calculated from the results obtained by Method IV, regarded as correct.

Fertilizer	Method I. P <sub>2</sub> O <sub>5</sub> p. c.	Method II. P <sub>2</sub> O <sub>5</sub> p. c.	Method III. P <sub>2</sub> O <sub>5</sub> p. c.	Method IV. P <sub>2</sub> O <sub>5</sub> p. c.
Superphosphate	10.88			10.50
“		9.49	9.38	9.37
Acid phosphate	14.65			14.20
“	15.20			14.75
Tankage	16.96			16.06
“		16.86		16.44
Bonemeal	22.20			21.60
“		21.83		21.51
Rock phosphate	25.15	24.96		24.45
“		28.93	28.32	28.39
Bone-ash	30.25		29.76	29.80
Bone-black			34.39	34.33
	Av. error 0.56 p. c.	Av. error 0.36 p. c.	Av. error 0.04 p. c.	

A study of these results demonstrates that the common method of precipitation as carried out under Methods I and II will invariably give results too high. In fertilizer work the error thus introduced will vary all the way from one or two-tenths of a per cent. when the solution is carefully neutralized and when it chances to be very dilute, up to six-tenths per cent. or even higher when the solution is more concentrated, or when molybdate of ammonia is present, or where the solution is strongly or even moderately ammoniacal when the magnesia mixture is added. This great variation of error undoubtedly explains many of the differences that have occurred in

the analyses of different chemists. The errors thus obtained clearly arise from the too sudden formation of the ammonia-magnesia phosphate precipitate, the precipitate carrying down with it extraneous matter, the error increasing, of course, when a large amount of molybdate of ammonia is present. When no foreign matter is present and the exact theoretical amount of magnesium chloride is contained in the solution, this sudden precipitation is not injurious, as is seen in Method IV. Method III when carried out as here described gives accurate results even in the presence of molybdate of ammonia. This method, as it does not necessitate any previous neutralization of the solution with hydrochloric acid, and requires but one precipitation is the one to be most highly recommended. Strong ammonia water should be added in liberal amount to the solution, and also employed in the washing.

The breaking of the filter paper and washing of the molybdate salt into the beaker, without filtering the solution cannot be recommended. However thoroughly the yellow salt may be washed, there is always a slight residue insoluble in ammonia water. A solution and filtration of the molybdic precipitate is therefore necessary in accurate work.

LABORATORY OF STILLWELL & GLADDING, NEW YORK.

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## ABSTRACTS.

Abstracts from *Journal of the Chemical Society*, London, by Arthur H. Elliott, Ph. B., F.C.S.

**On the Volumetric Estimation of Bismuth in the Form of Oxalate.** BY M. M. PATTISON MUIR, M. A., F. R. S. E. AND C. E. ROBBS, B. A. B. Sc. (Vol. XL., p. 1.)—Authors describe a process which depends upon the fact that potassium oxalate precipitates Bismuth as  $\text{Bi}_2(\text{C}_2\text{O}_4)_3 \cdot \text{K}_2\text{C}_2\text{O}_4$ . The method of procedure is as follows:—

A solution of the bismuth in the minimum quantity of nitric acid is evaporated till only enough acid remains to give a solution when hot; a large excess of glacial acetic acid is added and the whole transferred to a measuring flask, washing with acetic acid (1:1). Standard potassium oxalate is now added till there is twice as much of this salt present as there is bismuth oxide; the flask is filled to the mark with acetic acid (1:1) and after shaking and allowing to

settle, a portion of the fluid is filtered through a dry filter and the excess of potassium oxalate determined by a standard solution of permanganate.

The bismuth must first be separated from other heavy metals preferably as oxychloride. The double bismuth and potassium oxalate is soluble in potassium oxalate; but precipitates with excess of strong acetic acid.

**Action of Water on Bismuth Iodide. A Lecture Experiment.** BY M. M. PARRISON MUIR. (Vol. XL., p. 4.)—This experiment illustrates the influence of time, temperature, and mass, upon chemical change. Author pours solution of  $\text{BiI}_3$  in hydriodic acid, into cold water, water at  $90^\circ$ — $100^\circ$  C. and large quantity of cold water. In the first brown  $\text{BiI}_3$  is formed, in the second red  $\text{BiOI}$ , and small quantity of red  $\text{BiOI}$  in the third.

**Aluminium Alcohols. Part II. Their Products of Decomposition by Heat.** BY J. H. GLADSTONE, Ph. D., F. R. S., AND ALFRED TRIEBE, F. C. S. (Vol. XL., p. 5.)—This paper is a continuation of the highly interesting experiments of the Authors upon aluminium alcohols. The authors state that the general result of heating these alcohols is the formation of two sets of products, alumina and the alcohol and olefine, or alumina and the ether. They describe the products obtained by heating 1, aluminium phenylate, 2, aluminium para-cresylate, 3, aluminium thymolate; 4; aluminium beta-naphthylate, 5, aluminium gamma naphthylate. Among the products mentioned we note phenyl ether, cresyl-ether, metacresyl-ether, beta-naphthyl-ether. The paper is full of interesting facts and this abstract gives but a faint idea of the ground covered by the investigations.

**On the Action of Oxides on Salts. Part IV. Potassic Chlorate and Ferric Oxide.** BY EDMUND J. MILLS, D. Sc., F. R. S., AND GEORGE DONALD. (Vol. XL., p. 18.)—Experiments of authors go to prove that practically 3  $\text{Fe}_2\text{O}_3$  will expel  $\text{O}_{10}$ . And in conclusion they state "that the case of chemical change which we have under consideration presents nothing abnormal or peculiar in its features. From the carbonate, an oxide of carbon is the matter expelled; from the chlorate, an oxide of oxygen." The name *catalysis* ceases to have any reason for its existence.

In a supplementary note it is stated that the action of mangan-

ese dioxide upon potassic chlorate is the same as that of an oxide on a salt.

**Gamma and Beta-Amylan; Constituents of Some Cereals.** BY C. O'SULLIVAN. (Vol. XL., p. 24.) — Author describes two bodies of the same percentage composition as starch, one insoluble in water, and the other soluble. Barley contains 2 per cent. of gamma-amylan, and not more than 0.3 per cent. of beta-amylan. Wheat and rye contain 2.25 per cent. of beta-amylan, and not over 0.1—0.05 per cent. of gamma-amylan.

**Note on the Action of Ethyl-Chlorocarbonate on Benzene in presence of Aluminum Chloride.** BY EDWARD H. RENNIE, M. A., B. Sc. (Vol. XL., p. 33.) — Author states that ethyl-chlorocarbonate acts upon benzene in presence of alumina chloride in the cold producing carbonic anhydride, hydrochloric acid and *ethyl benzene*. Also states that ethyl-chlorocarbonate, by contact with aluminium chloride in the cold, yields carbonic anhydride and mono-chlorethene. When ethyl-chloroacetate is substituted for ethyl-chlorocarbonate, no reaction takes place.

**On Benzyl-Phenol and its Derivatives.** BY EDWARD H. RENNIE, M. A., B. Sc. (Vol. XL., p. 33.) — Author describes the preparation of the monosulphonic acid, potassium nitrobenzyl-phenol-sulphonate, potassium bromo-benzyl-phenol-sulphonate, and trinitro benzyl-phenol, and gives descriptions of their properties. Author appears to think that benzyl-phenol is a para derivative.

**On the Steeping of Barley.** BY EDMUND J. MILLS, D. Sc., F.R. S., AND J. PETTIGREW. (Vol. XL., p. 38.) This paper treats of the action of water, containing different quantities of calcium carbonate and calcium sulphate in solution, upon barley. In the case of calcium carbonate, as the latter diminishes the extract decreases, and the nitrogenous matters in the extract increase; but distilled water extracts less of the nitrogen compounds than 0.0224 per cent. solution of calcium carbonate. With calcic sulphate the total extract or nitrogen per unit of calcic salts is much greater than with the carbonate. The paper also contains an analysis of Burton water, and its effect upon the steeping of barley. It is shown that the extract decreases as the water is diluted with distilled water. Factitious waters were made containing some of the important constituents of the Burton water; and as a result of

steeping barley it was found that with calcic sulphate and sodic chloride, more extract and less nitrogenous matter was obtained than with Burton water; while with calcic carbonate and sodic chloride the extract is about the same and the nitrogen less than with Burton water. The general effect of a calcic solution is to keep back the nitrogen within the grain. "The special esteem in which the Burton water is held may therefore be due to its nitrate, which is well known to have a highly stimulant action in the germinating of malt—a process which demands much oxygen."

**Researches on the Relation of the Molecular Structure of Carbon Compounds to their Absorption Spectra.** By W. N. HARTLEY, F. R. S. E. (Vol. XL., p. 45.) PART VI.—*On the constitution of Pyridine, Picoline, Quinoline, and Cyanuric Acid.*

After experimenting on Hydrocyanic Acid, the author comes to the conclusion that: "The simple union of carbon to nitrogen does not cause selective absorption of the ultra violet rays." A number of diagrams from photographs of absorption spectra are given and comparison is made between the different spectra. Author thinks it probable that in benzene each carbon atom is united with three other carbons. A full idea of the paper cannot be given without the diagrams.

**On Peppermint Camphor (Menthol) and some of its Derivatives.** By R. W. ATKINSON, B. Sc., AND H. YOSHIDA. (Vol. XL., p. 49.) Authors describe Menthone  $C_{10}H_{18}O$ , give its vapor density as 77.45 and 76.69; it boils at  $206.3^{\circ}C$ , and its specific rotary power is  $[\alpha]_D^{20} = +21.16^{\circ}$ . Its specific gravity at different temperatures as well as its rate of expansion and molecular refraction are also given. Menthone  $C_{10}H_{18}$  is also described; it boils at  $167.4^{\circ}C$ , its specific gravity power is  $[\alpha]_D^{20} = +13.25^{\circ}$ . The specific gravity, rate of expansion and molecular refraction are also given.

**On the Composition of Pennant Grits in Contact with and at a Distance from Carbonaceous Deposits.** By EDWARD WETHERED, F. C. S., F. G. S. (Vol. XL.,) A comparison of a number of analyses of the grits that accompany coal seams. The author finds that these become more argillaceous when in contact with the coal, and assigns the cause to the carbonic acid given off from the decomposing vegetable matter, acting upon the freshly formed inorganic sediment; decomposing all silicates except silicate of alumina.

XXX.—ON THE PROPERTIES OF PURE METALLIC ALUMINUM.

By J. W. MALLET.

In view of the marked effect upon the properties of the individual metals, which is often produced by the presence of even very small quantities of foreign substances, metallic or non-metallic, it is very desirable that when really pure specimens can be had, prepared and tested with unusual care, their properties shall be noted and recorded. Comparatively few of the metals have probably been procured and examined in a state of the highest attainable purity, and comparatively few chemists have learned by personal experience how very difficult it is to get rid of the last detectable traces of foreign matter. This difficulty is especially great in the case of those metals least easily reduced to the metallic state, among which may be counted aluminum.

The writer, having expended much time and labor upon obtaining pure metallic aluminum to be used in his work\*, on the atomic weight of this element, succeeded in preparing enough for that purpose, and incidentally noted the following facts in regard to the metal—not forming a complete description of all its properties, physical and chemical—but yet deserving, perhaps, of being placed on record.

The crude (commercial) aluminum, used as the source of the pure metal, was found to contain:

Aluminum (by difference).....	96.89
Iron.....	1.84
Copper.....	Trace
Silicon.....	1.27
	<hr/>
	100.00

This was converted into bromide by treatment with liquid bromine, observing the precautions described in the paper (in Phil. Trans.) above referred to. A large quantity of the aluminum bromide—more than two kilogrammes altogether—was purified by careful fractional distillation, with due regulation of temperature, repeated a number of times, until the product was perfectly colorless, and on solution in water gave no evidence of the presence of

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\* Philosophical Transactions—Pt. III., 1880.

impurities, which were searchingly tested for. The reduction of the metal from this purified bromide was accomplished, but with much difficulty and great loss, by heating with pure sodium in a crucible made of, or lined with, a mixture of purified and dry alumina and sodium aluminate. The large globules of aluminum obtained were severally re-fused before the blowpipe on a support of alumina, and were then exposed for a short time to the action of hydrochloric acid on the surface, well washed with water, and dried. On solution of portions of the globules in pure hydrochloric acid, careful testing failed to show the presence of any of the impurities which might have been taken up from the materials and apparatus used. The entire absence of sodium was specially verified.

The color of this pure aluminum was perceptibly whiter than that of the commercial metal—on a cut surface very nearly pure tin-white, without bluish tinge, so far as could be judged from the small pieces examined. The lustre, too, was very much that of tin in a fresh, untarnished condition.

The metal was distinctly softer than before purification. Hence its fracture was not easily observed, but seemed to be very fine grained, with some appearance of fibrous silkiness. The malleability was undoubtedly improved, the metal yielding easily to the hammer, and bearing distortion well by flattening in two or three directions without cracking. It seemed to be sensibly less hardened by hammering than the ordinary metal of commerce.

The specific gravity was carefully determined at  $4^{\circ}$  C., and the mean of three closely concordant experimental results gave the number 2.583 as referred to water at the same temperature.

Hence the "atomic" volume = 10.45, near that of gold (10.19, if the sp. gr. be taken as 19.295, the value found by Roberts for the large trial-plate of fine gold of the English mint), and standing to that of iron nearly as 3:2 (the at. vol. of the latter being 7.11, if the sp. gr. be taken = 7.88, as found by Caron for pure iron fused in the oxy-hydrogen flame).

The specific heat was determined by means of Bunsen's ice calorimeter and found to be = .2253 as a mean for the range of temperature  $0^{\circ}$ — $100^{\circ}$  C., a number rather higher than those obtained by Regnault and Kopp. This number multiplied by 27.02, the atomic weight found in the experiments for which the metal was prepared, gives the product 6.09 as the atomic heat.

The quantity of pure material at command was not sufficient to



allow of any determination of the conductivity, though this would be an interesting point to examine, in view of the high conducting power of the commercial metal, and the probability that the value would be notably raised by the removal of impurities. An imperfect attempt was made to measure the expansion by change of temperature, using for the purpose determinations of specific gravity in water at widely different temperatures, and correcting for expansion of water and glass, but the quantity of material was too small to make the result trustworthy.

It was also attempted to estimate roughly the fusibility, by placing equally heavy beads of the pure and the commercial aluminum in front of a fine blowpipe jet, this jet being fixed in position and fed with air by a steady water blast. From the difference of distance at which the beads could be just fused it seemed probable that the pure metal was a little less fusible than that of commercial character.

Under the same conditions of heating the former seemed to oxidize on the surface with rather more ease than the latter. On the other hand, the pure metal seemed to present perceptibly greater resistance to the prolonged action of solvents—acids and alkalies—than the impure.

UNIV. OF VIRGINIA,

*July 15, 1882.*

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## CONTRIBUTIONS FROM THE LABORATORIES OF THE SCHOOL OF MINES, COLUMBIA COLLEGE.

### XXXI.—ON NITRO-SACCHAROSE.

By ARTHUR H. ELLIOTT, Ph. B., F. C. S.

This curious body has already been investigated by Sobrero in 1847,\* Schoenbein,† Ad & W. Knop‡ and others; and no definite conclusions as to its constitution appear to have been brought out by their labors. Having to make some experiments upon this body I thought it would not be uninteresting to give an account of some results which I obtained in the course of my work. These experiments include the action of a number of solvents, the action

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\* Comptes Rendus Vol. 24, p. 247.

† Pogg. Ann. Vol. 70, p. 100.

‡ Jour. fur prak. Chem, Vol. 56, p. 334.

of ammonium sulphhydrate, and also the action of a strong solution of ferrous chloride.

In the preparation of nitrosaccharose, pure crystals of cane sugar used and reduced to a powder fine enough to pass through a sieve with 100 holes to the linear inch, after which it was dried in a water-bath. A number of methods were used to nitrate the sugar, and a brief description of these may prove useful. In the first case the following method was used. 50 grms. of sulphuric acid (sp. gr. 1.84) and 25 grms. of nitric acid (sp. gr. 1.53) were mixed together, and the mixture cooled to  $15^{\circ}$  C.—25 grms. of the finely powdered sugar were now slowly stirred into the acid mixture, the temperature rose to  $30^{\circ}$  C., the mixture being constantly stirred for 15 minutes. The clot thus formed was thrown into cold water (about 1 litre) and the acid mixture was also added. The nitrosaccharose thus formed was collected together into one mass, and after washing and squeezing in plenty of cold water it weighed 24.4 grms.

The material thus obtained was a soft, silky looking, wax-like body, plastic, and adhering to the fingers when removed from water. At  $30^{\circ}$  C. it is soft enough to flow like melted pitch. It could not be washed free from acid. When heated on a water-bath it melted and began to decompose giving off red fumes. An attempt was made to neutralize it with potassic carbonate, but the action was very energetic and most of the material dissolved in water.

Another lot of nitrosaccharose was made using 25 grms. of sugar 400 grms. of sulphuric acid (sp. gr. 1.84) and 150 grms. of nitric acid (sp. gr. 1.53) following the same method of adding the sugar as mentioned before. The mixture had a temperature of  $15^{\circ}$  C. before adding the sugar and rose  $10^{\circ}$  C. during the experiment. In this case the acid mixture containing the clot of nitrosaccharose was poured into cold water and the mixture of acids and water became so hot that red fumes were evolved. The resulting mass after washing and squeezing weighed only 22 grms. The mass was melted on a water-bath under water, and while in this condition potassium carbonate was added and the whole well stirred. It was then washed from excess of alkalies and allowed to stand in water several days, changing the water daily. After a time it began to give an acid reaction, showing decomposition. It was washed with a little alcohol and allowed to dry.

A third lot of nitrosaccharose was made, using the same acids and sugar in the following proportions: sugar, 22 grms., sulphuric

acid, 176 grms., nitric acid, 66 grms. The sugar was added as usual, and the resulting mixture was poured into water in small portions, cooling the water after each addition; the nitrosaccharose thus obtained after washing and squeezing weighed 27 grms. It was washed with water and a weak solution of potassic carbonate.

The rest of the treatment was exactly like that followed in the second case.

Yet another quantity of nitrosaccharose was made, using a large quantity of sugar. Eight hundred grms. of sulphuric acid (sp. gr. 1.84), and 300 grms. of nitric acid (sp. gr. 1.53), were used with 95 grms. of sugar. When half the sugar had been added to the acid mixture, the whole was cooled, the rest of the sugar added and the completed mixture allowed to stand one hour with frequent stirring. The acids were now poured off the nitrosaccharose, and the latter, when washed and kneaded thoroughly with water, weighed 135 grms. When the mass was first removed from the acid mixture it had the consistence of soft butter, but as the washing and squeezing progressed it became more and more resinous and white. A further kneading in water hot enough to bear the hand in it, using two lots of about one litre each, and finally in water containing one per cent. of potassic carbonate, reduced the well-squeezed mass to 109 grms.

This last method appears to give the best result in the matter of washing, but the material slowly decomposes, the water in which it is kept becomes acid and smells faintly of hydrocyanic acid. Nevertheless the nitrosaccharose made by either of the methods changes so slowly that I have kept it under water for over a year without any great change in appearance or bulk, beyond the acid reaction mentioned above.

*Action of Solvents on Nitrosaccharose.*—The following solvents were used upon nitrosaccharose, both at ordinary temperature and also by heating in a water-bath:

Water—*Cold*, soluble; *hot*, soluble.

Absolute alcohol—*Cold* and *hot*, soluble.

Alcohol, 93 per cent.—Same as absolute.

Alcohol, 80 per cent.—*Cold*, slowly soluble; *hot*, soluble.

Alcohol, 50 per cent.—*Cold*, insoluble; *hot*, soluble.

Ether—*Cold* and *hot*, soluble.

Chloroform—*Cold* and *hot*, soluble.

Benzole—*Cold*, insoluble; *hot*, slowly soluble.

Carbon disulphide—*Cold*, insoluble; *hot*, insoluble.

Sulphuric acid (sp. gr. 1.84)—*Cold* and *hot*, soluble.

Acetic acid (glacial)—*Cold* and *hot*, soluble.

Turpentine—*Cold* and *hot*, insoluble.

Wood naphtha—*Cold* and *hot*, soluble.

Amylic alcohol—*Cold*, partly soluble; *hot*, soluble.

Carbolic acid—*Cold*, partly soluble; *hot*, soluble.

Nitrobenzole—*Cold* and *hot*, soluble.

Toluol—*Cold*, insoluble; *hot*, partly soluble.

Petroleum naphtha (71°–76° B'e)—*Cold* and *hot*, insoluble.

Astral oil—*Cold* and *hot*, insoluble.

Mineral sperm—*Cold* and *hot*, insoluble.

Olive oil—*Cold*, insoluble; *hot*, soluble.

Sperm oil—*Cold*, insoluble; *hot*, soluble.

Potassic hydrate (1 in 10)—*Cold*, insoluble; *hot*, partly soluble.

Ammonia hydrate—*Cold*, insoluble; *hot*, partly soluble.

Nitric acid (conc.)—*Cold* and *hot*, soluble.

Hydrochloric acid (conc.)—*Cold*, partly soluble; *hot*, slowly soluble.

Stannous chloride, decomposes it.

Ferrous sulphate, decomposes it.

Ferrous chloride, decomposes it.

Ammonia sulphhydrate, decomposes it.

Borax (5 per cent. solution)—*Cold* and *hot*, insoluble.

Acetic ether—*Cold* and *hot*, soluble.

Acetone—*Cold* and *hot*, soluble.

Glycerine—*Cold*, insoluble; *hot*, soluble.

Nitroglycerine—*Cold*, slowly soluble; *hot*, readily soluble.

While trying the action of these solvents the following facts were noticed. When heated with water it decomposes as it dissolves. On heating with turpentine a slightly yellow fluid is obtained. Carbolic acid is reddened, both cold and hot. Toluol, both cold and hot, gives a red color. With olive oil mutual decomposition takes place on heating. Sperm oil acts like olive oil. Potassic hydrate gives a brown solution when heated. Ammonic hydrate, even in the cold gives a yellowish-brown color. Nitric acid, decomposes it on heating giving red fumes. Hydrochloric acid, both cold and hot decomposes nitrosaccharose. Stannous chloride is decomposed giving a white precipitate, even in the cold. Ferrous sulphate, also chloride, decomposes nitrosaccharose, even in the cold, giving brown precipitates. Ammonic sulphhydrate, both hot and cold, decomposes this body, giving a liver colored solution, with separation of sulphur.

*Action of Ferrous Chloride upon Nitrosaccharose.*—Solution of ferrous chloride was prepared 10 c.c. of which contained 1.4 grms. of iron in the ferrous state. Nitrosaccharose was dissolved in 93 per cent. alcohol and the solution mixed with ferrous chloride solution mentioned above. After heating on a water-bath excess of potassic hydrate was added, the solution filtered and the precipitate washed. Two samples of this solution were taken; one was used to estimate glucose direct with Fehling's solution; the other was heated with acid to invert the sugar, and then, after neutralizing, the glucose was determined as in the first case. They both gave 11.7 per cent. of glucose on the original amount of nitrosaccharose taken, showing that, as might have been expected, heating with ferrous chloride solution inverts all the sugar in nitrosaccharose. This action should be compared with the reducing action of ammoniac sulphhydrate mentioned further on. It should be mentioned here that the ferrous chloride contained no free acid, since it was made by adding a large excess of iron to hydrochloric acid and heating till action ceased, a large quantity of iron remaining undissolved which was left in the solution to keep it saturated.

To determine the amount of oxidation produced by the nitrosaccharose upon the ferrous chloride, several experiments were made. In the first case the nitrosaccharose was treated with ferrous chloride and hydrochloric acid in an atmosphere of carbonic acid. The excess of ferrous chloride remaining after the action, was determined with standard solution of potassic dichromate. .705 gm. of nitrosaccharose oxidized .583 gm. of iron, which is equal to 30.6 per cent. of  $\text{NO}_3$  in nitrosaccharose. In another experiment the nitrosaccharose was dissolved in ether and the ether solution was shaken up with pure calcic carbonate. After filtering, the calcic carbonate was washed with ether and the filtrate and washings evaporated to dryness. The residue was washed with water and contained only a trace of calcic nitrate, hence nitrosaccharose contained little free acid. The water-washed residue was now dissolved in ether and treated with ferrous chloride solution and hydrochloric acid on a water bath, the excess of ferrous salt being determined with potassic dichromate as in previous experiment. .572 gm. of nitrosaccharose oxidized .645 gm. of iron which is equal to 41.6 per cent. of  $\text{NO}_3$  in nitrosaccharose. Another experiment in which the treatment with calcic carbonate was omitted and the ether solution treated at once with ferrous chloride and hydrochloric acid, gave the following results: .563 gm. of nitrosaccharose oxi-

dized .633 grm. of iron which is equal to 41.3 per cent. of  $\text{NO}_2$  in nitrosaccharose.

From these experiments it appears that the body under consideration acts as a nitrate and contains but a very small quantity of free acid when thoroughly washed and squeezed. In treating with ferrous chloride direct without solution in ether, some of the nitrosaccharose is decomposed without oxidizing the iron solution.

*Action of Ammonic Sulphhydrate upon Nitrosaccharose.*—For these experiments the nitrosaccharose was dissolved in 93 per cent. alcohol and treated with ammoniac sulphhydrate, which was made by saturating ammoniac hydrate with sulphuretted hydrogen. In each case a weighed quantity of nitrosaccharose was dissolved in the alcohol, and a large excess of ammoniac sulphhydrate was added to the solution. The mixture was then placed on a water bath and heated till a dry residue was left, which was treated with water, and filtered. The insoluble matter was examined and found to be sulphur. The solution was heated directly with Fehling's solution and gave only a trace of cuprous oxide, showing that the sugar had not been inverted during the reduction. This fact was established by several such experiments. Several experiments were now made by reducing the nitrosaccharose in alcoholic solution with ammoniac sulphhydrate, as before stated, and then inverting the sugar by heating two hours with a little sulphuric acid. By this means the nitrosaccharose gave 28.4, 28.7, 26.5, 24.9 per cent. of cane sugar. The first and second results are probably correct, as the third and fourth results gave solutions which were colored, apparently from formation of caramel from over-heating. The acid solution obtained by pouring the mixture of sulphuric and nitric acids, after treating the sugar, in water, was kept and a special examination was made for oxalic acid. For this purpose 50 c.c. of the diluted solution were neutralized with ammoniac hydrate and solution of calcic chloride added. After a time a precipitate was formed which proved to be calcic sulphate, but no calcic oxalate could be detected in it.

From a consideration of the foregoing experiments the following conclusions may be drawn : Ferrous chloride in presence of hydrochloric acid reduces nitrosaccharose and converts the sugar into glucose. Ammonic sulphhydrate reduces nitrosaccharose without inverting the sugar, or only to a very slight extent. In regard to the experimental determination of sugar and  $\text{NO}_2$  in nitrosaccharose: if we take the percentage of sugar found and add it to the

percentage of  $\text{NO}_3$  found by oxidizing ferrous chloride, we obtain the following results: The average of the first and second determinations of sugar by reducing with ammoniac sulphhydrate (28.4 and 28.7) gives 28.55 per cent. of cane sugar in nitrosaccharose. The average of two determinations of  $\text{NO}_3$  in nitrosaccharose (41.6 and 41.3) gives 41.45 per cent. of  $\text{NO}_3$  in that body. Adding these together we have 70.00 per cent. as a total; the balance of the percentage consists of water, some free acids, and probably other compounds resulting from the action of the acids upon the sugar. Calculating the determined constituents to per cent., we have cane sugar, 40.78, to  $\text{NO}_3$  59.21, or a ratio of 1 molecule of cane sugar to 8 molecules of  $\text{NO}_3$  (exactly 1 to 8.009). From these figures it would appear that nitrosaccharose is formed by replacing eight atoms of hydrogen in a molecule of sugar by eight molecules of  $\text{NO}_3$ , giving the formula  $\text{C}_{12} \text{H}_{14} (\text{NO}_3)_8 \text{O}_{11}$ . Assuming that this is the correct formula for nitrosaccharose, the theoretical percentage yield of its constituents would be sugar 41.20,  $\text{NO}_3$  59.75. When we compare these figures with those actually obtained above, and remember that the methods used are not capable of great exactness in results, it appears most probable that the above is the true formula for nitrosaccharose.

### XXXII.—NOTES ON BONE OIL.

BY ARTHUR H. ELLIOTT, Ph. B., F. C. S., AND FERDINAND SANDS, A. B., Ph. B.

During the Spring of 1881, Messrs. C. P. Sawyer and W. W. Share, two students of the class of 1881, of the School of Mines, at the suggestion of Prof. C. F. Chandler, undertook to investigate bone oil, and repeat some of the work of Anderson,\* C. G. Williams,† Vohl,‡ Church, and Owen§, Schwanert,|| Unverdorben¶ and Runge.\*\*

\**Phil. Mag.*, (3) vol. xxxiii. p. 174; (4) vol. ii. p. 457; (4) vol. ix pp. 145 and 214. *Trans. Roy. Soc. Edin.*, vol. xvi. p. 4; vol. xx. (2) p. 247; vol. xxi. (1) p. 219; vol. xxi. (4) p. 571.

†*Chem. Soc. Jour.*, (Lon.), vol. vii. p. 97. *Proc. Roy. Soc.*, vol. xiii. p. 305; vol. xii. p. 311.

‡*Jahresb.*, 1859, p. 742.

§*Jahresb.*, 1850, p. 359. *Phil. Mag.*, (4) vol. xx. p. 110.

||*Am. Ch. Pharm.*, vol. cxx. p. 279.

¶*Pogg. Ann.*, vol. xi. pp. 59, 67; vol. viii. pp. 258, 480.

\*\**Pogg. Ann.*, vol. xxxi. pp. 65, 67. *Chem. Gaz.*, 1855, p. 308.



For this purpose they took five barrels of bone oil and submitted it to distillation, a process whose disagreeable nature none can appreciate but those who have had the task. These hard working gentlemen succeeded in obtaining a number of interesting results from the various distillates, and it is to be hoped that some day they will publish an account of them. From the five barrels of bone oil they obtained (after losing about one and a half barrels by foaming) about ten gallons of light oil and water. On treating this light oil with sulphuric acid they obtained a reddish gelatinous mass which they placed on one side, and continued their work upon other products of the bone oil.

During last winter we thought it would be interesting to find out the character of the red gelatinous body, mentioned above, obtained by treating the light oil from bone oil with sulphuric acid. We therefore took some of the distillate obtained by Messrs. Sawyer and Share and treated it with sulphuric acid. Two litres of this distillate, were mixed with about twice the volume of water, 500 c. c. of concentrated sulphuric acid were added with constant stirring, and the whole allowed to stand twenty-four hours. At the end of this time a heavy granular reddish-yellow precipitate had separated, which was strained out through muslin and squeezed in a screw press to remove acid and oil. It was thoroughly washed with water, soaking many hours, between each new addition of wash water, and it gradually assumed a darker color, finally becoming a dark brownish-black. The liquid strained from the precipitate obtained above, and containing oil, was again treated with 500 c.c. more of concentrated sulphuric acid, well agitated, and allowed to stand twenty-four hours. Another quantity of the reddish yellow precipitate was found and separated as before; it also turned brownish-black after a time. The strainings from the second precipitate were treated with sulphuric acid, and more precipitate was obtained. A fourth treatment of the strainings gave but a little of the precipitate and the treatment was discontinued. During this treatment the oil became smaller in quantity and at last there was practically nothing left from these operations but the various precipitates and the strongly acid fluid with a little oil floating on the surface.

The original light oil had a specific gravity of 0.8354 at about 20°C. and boiled at about 120°C., at which temperature it is distilled very rapidly. This oil does not mix with water and is soluble in alcohol.



The brownish-black precipitates obtained above were all washed with ether, alcohol, and finally with hot water, and dried in a water bath. As various quantities of acid were added to the successive strainings, the precipitate became more densely gelatinous and of the consistence of leather that has been burned. But by treating with ether, alcohol, and hot water, it was found that all the precipitates could be reduced to the same black powder. This black powder was heated with the following solvents without effect: Alcohol, methyl alcohol, turpentine, glacial acetic acid, sulphuric acid, petroleum-naphtha, phenol, and glycerine, ether, chloroform, amyl alcohol, nitrobenzol, acetic ether, acetone, toluol, and carbon disulphide. If fuming nitric acid is dropped upon it violent deflagration takes place; but by pouring an excess of this acid upon the black powder, no action takes place immediately; only after long standing complete solution takes place. This solution, when diluted and excess of ammoniac hydrate added, gives a bright scarlet solution, but no precipitate.

We tried the action of these solvents in the hope of being able to crystallize this body, and finally we gave up the idea. We therefore treated it again with ether, alcohol and hot water, which gave us colored solutions in each case. On evaporating these solutions we find that only trifling quantities of oily matters were left; which, in the case of the ether solution, itself of a dark orange color, gave a slight green fluorescence before evaporation.

After careful purification as above stated, an analysis of the powder was made. Much trouble was encountered in determining the carbon and hydrogen, owing to the fact that the body was found to contain considerable sulphur. But we finally succeeded in getting duplicate analyses that were satisfactory. For this purpose we mixed the black powder with a large quantity of fused lead chromate in fine powder; the gases were then made to pass over granulated copper oxide and about four inches of clean copper gauze recently reduced in hydrogen.

The nitrogen was determined by heating with soda-lime, and gave very satisfactory results.

In the case of the sulphur we had another set of failures; the fine condition of the powder causing most violent action, with various oxidizing agents. We finally succeeded in getting fair results by fusing with a mixture of twelve parts of mixed carbonates (7 of potassic carbonate to 5 of sodic carbonate), and three parts of potassic nitrate, to one part of the powder. By this means, using

a moderate heat. We succeeded in getting a quiet fusion with very gentle delagration. Care must be taken that the fusion mixture is dry and finely powdered, and that the black powder is very intimately mixed before heating for fusion.

We made many determinations of the various constituents of this curious body, many of them approaching the results given below, which are those obtained after considerable experimenting on the various methods used for its analysis.

The following is the percentage composition of the brownish-black powder resulting from the change of the orange-red precipitate obtained by heating the bone-oil distillate with sulphuric acid:

	I	II.
Carbon.....	59.56	59.44
Hydrogen .....	6.28	6.22
Nitrogen .....	11.90	11.90
Sulphur .....	3.92	4.15

Anderson and Schwanert both analyzed this body, but no mention is made of the presence of sulphur in it. Anderson's figures are 71.98 carbon, 6.88 hydrogen, 13.59 nitrogen; while Schwanert, who made the body from carbopyrrolic acid, found smaller quantities of carbon and nitrogen. We make these statements since we believe this body is what is generally known as "pyrrol red," and formally examined by Anderson and Schwanert with the above results. Wiedel and Ciamician\* have recently worked upon this body, and have not been able to obtain it of constant composition, since they examined it in the red condition, when it changes very rapidly. The last authors do not mention the presence of sulphur in this body, and we have searched in vain for such a record. We are led to believe that pyrrol red is a compound of sulphuric acid (or hydrochloric acid when produced by that agent), with the light oil obtained from bone-oil, but cannot at present give the constitution of the body, as our work is incomplete. We would mention that by boiling with strong solution of potassic hydrate no action takes place and no perceptible quantity of ammonia is evolved; but by fusing with solid potassic hydrate after evaporation of the mixture of the black body with solution of potassic hydrate, we have obtained an oily distillate that appears very much like the original oil of the bone-oil distillate which we started with. At present we have not obtained enough of this material to examine its character.

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\*Berichte xiii., 65-85.

We have given these notes to call attention to our work and to see if others have not perhaps already arrived at similar conclusions. We are well aware that many others have labored hard in this field, but we have done our work with every endeavor to arrive at facts, and hope to be able to continue the investigation the coming winter, when we shall prepare this body in other ways and analyze the oil obtained by fusing with potassic hydrate.

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### XXXIII.—ON THE COMPOSITION OF ELEPHANT'S MILK.

BY CHARLES A. DOREMUS, M. D., PH.D.

In the spring of 1881 an opportunity presented itself for obtaining several specimens of milk from the elephant Hebe. The analysis of these samples were reported in Vol. III of the journal of the society. The exceptional character of the milk made it desirable to obtain further specimens. At the time the first investigation was undertaken two other female elephants were supposed to be with young. Such proved to be the case with one only.

The elephant Queen gave birth to a female calf at Bridgeport, Connecticut, on February 22, 1882, and the new baby was named after its birthplace. My intention was to collect some of the first milk secreted, but, though unsuccessful in this, I procured a quantity about thirty-six hours after the birth of the calf. This milk still possessed the qualities of colostrum.

The colostrum collected presented the appearance of thin milk. On standing it separated into three layers—the cream uppermost, then the usual blue layer, and at the bottom a yellow stratum composed mostly of mucous cells held together in a glairy fluid. These cells were uniform in size and in comparison with the oil globules not of that relative size usual in colostrum corpuscles. Of those peculiar bodies there were indeed few, unless these smaller cells represented them. The fat globules were not uniform in size, nor did they present that peculiar aggregation noticed in the milk from the elephant Hebe, or later that of Queen.

As the presence of albumen is one of the characteristics of colostrum, an attempt was made to determine its quantity, as well as the quantity of casein. The task proved difficult. At first Hoppe-Seyler's method was adopted, viz., that of acidulating the milk, greatly diluted, nearly to the point of coagulating the casein and then completing the reaction by saturating the liquid with  $\text{CO}_2$ . A flocculent

deposit was obtained after very long standing, but though the test was repeated several times no complete filtration could be effected. Qualitatively the experiment was successful ; the presence of albumen was readily detected in the filtrate on boiling.

As Tolmatscheff\* claims that Hoppe-Seyler's method proved unsuccessful in his hands when applied to milk from women and advises the use of magnesium sulphate to saturation to precipitate casein, and alcohol to precipitate casein and albumen together, experiments were made to test its value in this instance.

By the use of magnesium sulphate the casein was separated, but the milk became so thick and mucilaginous that though left to filter during two days so viscous a mass remained on the filter that all attempts to wash it with magnesium sulphate solution proved futile. The filtrate, what there was of it, was clear and gave a coagulum when heated. The coagulum seemed more copious than that in the filtrates by Hoppe-Seyler's method, but this may have been due to the greater concentration of the liquid.

Not being able to separate the casein by these methods in such form as to weigh it, 20 cc. of the milk were treated by Hoppe-Seyler's method and the albumen determined in 100 cc. of the filtrate. The combined percentages of casein and albumen were then ascertained, which, less the percentage of albumen already found, gave that of the casein. Alcohol proved to be the best precipitant for casein and albumen. The casein separated in fine flocks, carrying albumen and fat with it. The latter was subsequently removed by ether. The liquid filtered nicely, and the precipitate could be thoroughly washed on the filter. The usual method of precipitating albumen and casein by heat and acetic acid gave a precipitate that could not be separated by filtration. Three specimens of milk were collected. Most of the determinations were duplicated. The methods of analysis was the same as that followed in the case of the milk from the elephant Hebe, except as regards albumen and casein. It was not found necessary to dry the milk in vacuo. Once, with the colostrum, sand was mixed with the milk to aid dessication.

The sugar when determined by Fehling's solution volumetrically in the colostrum and No. II. was too low, but estimated gravimetrically in No. III. it agreed closely with the figures in the table. In No. I. the milk treated by alcohol and filtered left on evaporation a residue, less ash, equal to 8.0101 per cent. of sugar.

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\*v. Gorup-Besanez Physiologische Chemie, pp. 418 and 420.

	L	II.	III.	IV.
	Feb. 24th.	April 9th.	April 22d.	Milk
	Morning.	Afternoon.	Afternoon.	from Hebe.
Quantity.....	335 cc.	420 cc.	120 cc.	72 cc.
Cream.....	7.7 vol. %	7.7	33	62
Reaction.	Alkaline.	Slightly Acid.	Neutral.	Slightly Acid.
Sp. gr. at 60° F....	1,036	1,036	1,028	1,023.7
In 100 Parts by Weight.				
Water.....	86.5930	82.2390	76.9930	66.697
Solids.....	13.4070	17.7610	23.0070	33.303
Fat.....	3.0100	7.0576	12.6175	22.070
Solids not fat....	10.3970	10.7034	10.3895	11.233
Casein .....	2.0676	5.7060	5.7590	3.212
Albumen.....	0.3455			
Sugar*.....	7.6533	4.5494	4.1510	7.392
Ash.....	0.3306	0.4480	0.4795	0.629
Taste.....	Disagreeable	Much improved.	Excellent.	Excellent.
Odor... ..	Unpleasant	Pleasant	Pleasant	Pleasant
Color.....	{ Yellowish white	White and opaque	White and opaque	White and opaque
Consistency.....	{ Yellow de- posit, other- wise fluid	Very fluid	Fluid	Fluid

Specimen No. III. was drawn after the elephant had been exhibited in the circus circus-ring and while the calf was nursing at the other teat. The calf had not nursed for some hours. Of all it approaches nearest in composition to the milk from Hebe. The solids not fat, remain constant throughout, while the fat varies considerably. Apparatus to serve as a breast pump was constructed in order to collect the milk from one teat or both during say half a day, in order to gain a knowledge of the quantity secreted daily, but the plan unfortunately could not be carried out. “Bridgeport” grew quite as rapidly as the other “baby,” more than doubling her weight during the period of these experiments.

The character of the fat in the last specimen was like that obtained from Hebe, the size and aggregation of the globules and the odor of the fat after extraction being the same. During the last month's of Hebe's period of lactation the employees about the circus frequently partook of her milk and found it most palatable.

\*By difference.

# REVIEW OF PAPERS ON ANALYTICAL CHEMISTRY (IN-ORGANIC) WHICH HAVE APPEARED DURING THE YEAR 1882.

By E. WALLER, PH. D.

A modification of the apparatus for the absolute DETERMINATION OF NITROGEN, invented by S. W. Johnson and E. H. Jenkins, is described by *T. S. Gladding* (*Am. Chem. Jour.*, IV., 42).

According to *A. Guyard* (*Chem. News*, XLV. 159), ALL ACIDS AND OXIDES OF NITROGEN are converted into ammonia by combustion with marsh gas and soda lime. The mixture is made in the proportion of 75 grs. of sodium acetate with  $1\frac{1}{2}$  oz. of soda lime, and the combustion conducted in the ordinary manner. The tube is charged first with about one-quarter of the above mixture, then the remainder mixed with 5 to 7 grms. of the substance to be examined is added, and finally pure soda lime.

The accuracy of the results thus obtained is disputed by *J. Ruffle*, (*Chem. News*, XLV., 186).

In experimenting on methods for the DETERMINATION OF NITROUS OXIDE, *W. Hempel* (*Ber. D. Chem. Ges.*, XV., 903 *et seq.*) finds it extremely difficult to obtain absolutely pure  $N_2O$ . The purest gas obtained contained 98.8%. He finds the best method of estimating the gas to be by explosion with hydrogen in an apparatus specially designed for the purpose, which is carefully described.

100 vols.  $N_2O$  with 460 vols. H was not explosive, while 100  $N_2O$  with 116 vols. H caused such a violent explosion as to shatter the apparatus. The mixture therefore must be made within these limits.

Alkaline solutions of potassium permanganates or of chromic acid are recommended by *C. Boehmer* (*Fres. Zeits. Anal. Chem.*, XXI., 212,) as ABSORBENTS FOR  $N_2O_2$  in analytical processes.

To determine the amounts of sodium SULPHIDE, SULPHITE AND THIOSULPHATE in the same solution, *G. E. Davis* (*Jour. Soc. Chem. Ind.*, I., 89), determines, 1. The amount of iodine absorbed by a measured quantity of the solution, by converting the alkalies present into bicarbonates with carbonic acid water and titrating with iodine solution. 2. The amount of sulphide and thiosulphate present; by adding  $SrCl_2$  in excess, digesting for about half an hour at  $65^\circ$  to  $70^\circ$  C., filtering off the strontium sulphite, washing with air free from water, adding carbonic acid water to the filtrate, and titrating with iodine solution. 3. The amount of thiosulphate

present ; by separating the sulphite as in 2, and then separating the sulphide by digestion for half an hour with  $\text{CdCO}_3$ , filtering and titrating the filtrate with the same precautions as before. A correction must be applied to the results before calculating from the data obtained. It has been found that 100 cc. of a solution of  $\text{SrSO}_3$  saturated at 65 to 70° C., consumes 0.3 cc. decinormal iodine, and the same bulk saturated at 12.7° C. consumes 0.16 cc. decinormal iodine solution. The bulk and temperature of the filtrates must therefore be taken.

To obtain the SULPHUR IN PYRITES in a form for estimation as sulphate, *Fr. Bockmann* (*Fres. Zeits. Anal. Chem.*, XXI. 90), fuses 0.5 gms. with 25 gms. of a mixture containing 6 parts  $\text{Na}_2\text{CO}_3$  to 1 part  $\text{KClO}_3$ .

In estimating CHLORINE GRAVIMETRICALLY, *D. Lindo* (*Chem. News*, XIV., 193) filters the  $\text{Ag Cl}$  through a Gooch filter and dries for half an hour at 140 to 150° C.

In the indirect estimation of CHLORINE AND BROMINE, *L. P. Kinnicutt* (*Am. Chem. Jour.*, IV., 22), after weighing the mixed silver salts in a platinum crucible, reduces to metallic silver by a battery, the crucible being made the positive electrode of the circuit.

The sanitary EXAMINATION OF GASES ESCAPING FROM CHEMICAL WORKS forms the subject of a paper by *W. J. Lovett* (*Jour. Soc. Chem. Ind.*, I., 210). For *nitrous compounds* the gases are brought into contact (by agitation) with hydrogen peroxide and water, the solution neutralized with pure soda solution (free from nitrates) evaporated to very small bulk, and after removing carbonic acid by a drop of sulphuric acid, using a Davis nitrometer tube with conc.  $\text{H}_2\text{SO}_4$  and mercury, the volume of  $\text{N}_2\text{O}_2$  being measured. For *sulphurous acid* the gases may be passed through a standard solution of potassium permanganate. For *oxygen*, the gas is caught in a U tube, one limb of which is sealed and graduated so as to form an eudiometer, and oxygen is absorbed by potassic hydrate and pyrogallie acid. For *sulphuretted hydrogen* the gas may be passed through  $\text{Pb}(\text{NO}_3)_2$ , and the precipitate converted into sulphate and weighed, or the excess of lead remaining in the solution titrated with potassium dichromate. For *chlorine* the gases are passed through  $\text{KI}$  solution, and the iodine set free titrated with sodium thiosulphate. To distinguish between the amounts of *free and combined chlorine* in such escapes, *G. E. Davis* (loc. cit. p. 214) passes the gases through  $\text{H}_2\text{S}$  water. The solution thus obtained is halved. One half is neutralized with  $\text{Mg CO}_3$ , and then titrated with  $\text{Ag NO}_3$ ,



giving total chlorine ; the other half evaporated to neutrality without any addition and titrated, giving combined chlorine.

To determine the amount of OZONE IN THE AIR, *D. Tommasi* (*Les Mondes*, I. [3] 123), draws a measured quantity through a tube having two branches. The air passing through one branch is drawn directly through a standard solution of sodium arsenite ; that passing through the other branch is first passed over red-hot platinum sponge before passing into a solution of sodium arsenite. The solutions are then titrated with potassium permanganate, and the difference of the results in the two solutions serves as a basis for the calculation of the ozone present, as in the second solution the ozone has been destroyed by the ignited platinum sponge.

Experiments on potassium permanganate in relation to its application for the EXAMINATION OF POTABLE WATERS, have been made by *A. Dupre* (*Analyst* VII. 1). Water acidified with sulphuric acid when absolutely pure, does not decompose permanganate in a closed flask at 80° F. In standardizing the permanganate for Tidy's test the operation should be performed in a closed flask. With waters for domestic use which are comparatively free from organic matter, the degree of temperature and the length of time for the action of the permanganate as well as the quantity of permanganate, make but little difference in the results obtained. With less pure waters the differences between the 3 hour and 4 hour test were very decided. On using phosphoric acid instead of sulphuric in Tidy's test, results much lower were obtained. The addition of sulphuric acid and boiling before adding permanganate, increases the amounts of oxygen absorbed. This increase was very marked when the test was tried on pure water, to which cane sugar, starch, or urine had been added, but was much less marked when gelatine or egg albumen was used. *C. W. Marsh* (*Am. Chem. Jour.* IV. 188) finds that in many waters examined, the distillate obtained by distilling with sodium carbonate, affords more ammonia (determined by the Nessler) on redistilling with alkaline potassium permanganate than if the Nessler test is directly applied to it.

The ORGANIC NITROGEN in waters, beer, etc., is estimated by *W. Bettel* (*Chem. News*, XLV. 38), in one operation by distilling from a copper flask in a current of hydrogen with pure soda (from sodium). The use of the copper flask admits of distillation to complete dryness, and ignition of the residue. At the close of the operation to break up cyanides, &c., which may have formed, a little potassium permanganate is added, and heat again applied. Nitrates, if



present, are previously converted into ammonia by the action of the zinc-copper couple. The distillate contains all the nitrogen in the form of ammonia.

To convert NITRATES AND NITRITES IN WATER into ammonia, *F. P. Perkins* (*Analyst* VII. 66,) proposes the use of metallic magnesium aided perhaps by the addition of some Na Cl. *J. B. Kinnear* (*Chem. News*, XLVI. 33) finds that zinc with a dilute acid will effect the reduction in about ten minutes, provided that there are about 5,000 parts of liquid present for every part of nitric nitrogen. The rapidity of the reduction depends somewhat upon the amount of surface of zinc exposed.

Among delicate TESTS FOR NITRITES in water, *A. Jorissen* (*Fres. Zeits Anal. Chem.* XXI. 210) proposes a solution containing 0.01 gm. fuchsin in 100cc. of glacial acetic acid, which gives a change of color, passing through blue and green to reddish yellow. (Noted by *Vogel Jour. Prakt. Chem.* XCIV. 457), while *E. W. Davy* (*Chem News* XLVI. 1) recommends for the same purpose acidifying the solution, adding a few cc. of gallic acid and boiling. The presence of nitrites at once gives a tint to the water similar to that of the Nessler test with ammonia. The reaction is stated to be :



tanno-melanic acid being formed.

The COLOR METER FOR WATERS used by Messrs. *Crookes, Odling, and Tidy* in their examination of the London water (*Report to Local Government Board for 1881*) is described (*Chem. News* XLV. 170). The two-foot tube is used. For comparison colors, two wedges, one brown, the other blue, are used, which are slid over one another until a tint corresponding to that in the water is obtained. The solutions contain respectively :

*Brown*, 0.7 gm. metallic iron as ferric chloride  
0.3 " " cobalt as chloride,

and a slight excess of free acid per litre.

*Blue*, 5 gms. pure crystallized cupric acid sulphate per litre. The results are recorded in figures representing the thickness in millimetres of the solutions required to simulate the color of the water thus : Brown : Blue, 20 : 10.

Regarding the examination of fertilizers for REVERTED OR RETROGRADE PHOSPHATES, *C. Richardson* (*Am. Chem. Jour.* IV. 183) finds that the use of ammonium oxalate for their extraction gives variable results depending upon the time and temperature of diges-

tion, the degree of concentration of the solution, &c. In this connection see also results of *T. S. Gladding* (this *Journal*, p 113). *F. J. Lloyd* (*Jour. Lond. Chem. Soc.* XLI. 306) triturates 0.5 gm. of the fertilizer with 50cc. of a solution containing 30 per cent. citric acid, and enough ammonia to render it strongly alkaline. The mixture is made up to 100cc. and allowed to stand over night at the temperature of the laboratory. In 50cc. of the clear solution the lime is separated by acidifying with acetic acid and adding ammonium oxalate, and in the filtrate the phosphoric acid is determined by the use of magnesia mixture.

As to VOLUMETRIC processes for PHOSPHORIC ACID, *C. Mohr* (*Fres. Zeits. Anal. Chem.* XXI. 216) shows by a series of experiments that even small amounts of ammonia salts have a marked effect on the results obtained by the uranium titration, while *H. Pemberton* (*Jour. Franklin Inst.* CXIII. 184). proposes the use of a solution of neutral ammonium molybdate containing 89.543 gms. of the salt per litre (1cc=0.003 gm.  $P_2O_5$ ). For titration 100 to 125cc. of the solution is taken [which should not contain over 0.1 gm.  $P_2O_5$ ] 10 gms of ammonium nitrate, and 2cc. of nitric acid [sp. Gr. 1.14] are added and the solution is heated to 140° F. or over. The end reaction is the absence of further precipitation. Toward the close of the titration portions of the solution have to be filtered to give opportunity to observe the end reaction. If the titration is overrun, a measured quantity of  $Na_2HPO_4$  solution of known strength is added and the titration resumed. From the burette reading 0.5cc. is to be deducted for the effect of the nitric acid added. Silica and organic matter cause erroneous results when present, while chlorides, iron [to the extent of 0.1 to 0.3 gm.] &c., do not.

In the EXAMINATION OF TITANIFEROUS IRON ORES, *T. M. Drown and P. W. Shimer* (*Am. Chem. Jour.* IV. 1), treat the ore (2 to 5 grms.) with 50cc. of H Cl. (Sp. Gr. 1.12) evaporate to dryness and heat in the air bath for an hour, add 50cc more H Cl., heat and filter; turbidity in the washings is prevented by the use of ammonium nitrate. The phosphoric acid is extracted from the residue by fusion with sodium carbonate, extracting with water, evaporating to dryness with nitric acid to remove silica, &c. Molybdate mixture may then be added to the solution. In the first solution (obtained by H Cl.) the H Cl. is removed by evaporation with nitric acid, when after partially neutralizing, molybdate solution is added. This last molybdate precipitate requires purifying by dissolving in ammonia, treating the undissolved residue with nitric acid and pre-

precipitating that solution with more molybdate. After obtaining all the ammoniacal solutions of the phospho molybdate free from silica, titanio acid, &c., they are united and precipitated as usual with magnesia mixture.

For TITANIC ACID these gentlemen decompose the ore by fusion with  $KHSO_4$  and after neutralizing and reducing with  $SO_2$  precipitate by boiling as usual. *P. T. Austen* and *F. A. Wilber* (*Am. Chem. Jour.* IV, 211) find that the addition of 50 to 60cc. of acetic acid aid materially in the precipitation of the titanio acid, giving good results on boiling, even when chlorides are present. The last named decompose the ore by fusion with alkaline carbonates and treating with  $HCl$ , evaporating, &c.

For the *determination* of CARBON AND SILICON IN IRON, STEEL, etc., *F. Watts* (*Chem. News*, XLV., 279) gives the preference to a modification of Wohler's method.—Volatilization of the iron in a stream of  $Cl$  gas and making a combustion of the carbon remaining. Two operations are required for each analysis. In the first after volatilizing the iron in a current of *dry* chlorine free from oxygen, the residue is submitted to combustion, and the *total carbon* is thus determined. In the second the residue (after volatilizing iron) is weighed, giving *total carbon and slag*, while the gases from this operation are passed into water, where the  $SiCl_4$  is decomposed, and by boiling, evaporating to dryness, etc., the  $SiO_2$  corresponding to *combined* silicon is obtained.

SODA COMPOUNDS may be estimated in potassium carbonate, according to *A. Van Hasselt* (*Jour. Soc. Chem. Ind.* I, 203), by treating a weighed quantity (converted into chloride) with  $HCl$ . (Sp. Gr. 1.189) saturated with  $NaCl$ .

To estimate CAUSTIC ALKALI IN PRESENCE OF CARBONATE, *R. B. Warder*, (*Sci. Proc. Ohio Mech. Inst.* I, 45) uses a process based upon the fact that phenolphthalein is colorless in the presence of alkaline bicarbonates. On titrating a very dilute solution of the sample (cold) until the phenol pthalein which has been added imparts no color to the solution, all the caustic and half of the carbonated alkali is neutralized, giving a result A. Then by boiling and titrating the remainder of the alkali is neutralized; result B.  $A+B$  measures total alkalinity, and  $A-B$  measures caustic alkali

To detect and separate ALKALIES IN ARGENTIC NITRATE. *M. Stolba* (*Jour. de Pharm. et de Chim.*) uses hydrofluosilicic acid. By the use of phenol pthalein *M. Richter* (*Fres Zeits. Anal. Chem.*, XXI, 205) determines acidimetrically the CHROMIC ACID IN

POTASSIC DICHROMATE. Many other indicators cannot be used on account of the oxidizing action of the chromic acid upon them.

By fusing hydrorufigallic acid with potassa a NEW INDICATOR for alkalimetric titration is made by *J. Oser and W. Kalmann* (*Monats hefte f. Chemie*, II, 50). It dissolves in alkalies with an olivegreen tint—a slight excess of alkali gives carmine red, changed to yellow by free mineral acids. It is unaffected by  $\text{CO}_2$ .

To DETERMINE ZINC in its ores, *A. Millot* (*Bull. Soc. Chim.*, XXXVII, 339) recommends dissolving with  $\text{HCl}$  and  $\text{KClO}_3$ , evaporating, adding ammonia and ammonium carbonate, diluting the filtrate therefrom, and in an aliquot portion of this filtrate, adding a little  $\text{K Cy}$  and precipitating by the battery. The addition of ammonium acetate prevents the corrosion of the electrodes.

In TITRATING ZINC by Fahlberg's method manganese must be first separated to obtain correct results. *R. W. Mahon* (*Am. Chem. Jour.*, IV, 53). In titrating with  $\text{Na}_2\text{S}$ , the use of paper soaked in thallium solution is recommended by *M. Schröder* (*Berg. u. Huttenmann, Zeit.* 1882, p. 4) as an indicator. If a slight excess of ammonia is present the thallium salt is unaffected by  $\text{Zn S}$ .

To separate NICKEL FROM COBALT *G. Delvaux* (*Comptes Rendus*) dissolves in aqua regia with excess of  $\text{HCl}$ —adds ammonia in excess, then potassium permanganate until the rose color holds for some time. Solution of pure  $\text{KHO}$  will then precipitate nickel (with manganese if present), leaving cobalt in solution. In case small amounts of nickel and large amounts of cobalt are present *A. Jorissen* (*Fres. Zeits. Anal. Chem.*, XXI, 208) precipitates with  $\text{NaHO}$ , adds bromine, then a few cc. of  $\text{K Cy}$ , and agitates in the cold. The cobalt is dissolved while the nickel remains in the precipitate.

*A. Carnot* (*Bull. Soc. Chim.*, XXXVII, 482) ESTIMATES CHROMIUM (as base) by adding sodium acetate to the solution slightly acidified, adding an alkaline phosphate and boiling. The presence of oxalates must be avoided. The precipitate dried at  $100^\circ$  contains  $6\text{ H}_2\text{O}$ . Ignited it consists of  $\text{Cr}_2(\text{PO}_4)_2$ , containing 51.86 per cent.  $\text{Cr}_2\text{O}_3$ .

In the examination of iron ores, etc., containing chromium error may be caused, according to *B. Reinitzer* (*Monats hefte f. Chem.*, 1882, p. 249), by the chromium preventing the precipitation of some of the iron and alumina on boiling the solution after neutralizing and adding sodium acetate.

For the detection of IMPURITIES IN COPPER several chemists communicate their methods—

For ARSENIC *J. Pattinson* (*Newcastle-on-Tyne Chem. Soc.*, Feb. 1882) dissolves in nitric acid; then by adding sufficient soda solution to a little more than neutralize the free acid present, the arsenic is precipitated as copper arsenate; after standing for half an hour with frequent stirring the precipitate is filtered and washed cold. The arsenic is thus concentrated in the precipitate. *A. H. Sexton* (*Chem. News*, XLV, 255) prefers to precipitate the arsenic from the nitric acid solution (by adding ferric nitrate, neutralizing, adding sodium acetate and boiling) as basic acetate. The precipitate is dissolved in acid, precipitated by  $\text{H}_2\text{S}$ , oxidized to arsenic acid, and determined as the magnesia compound.

For CADMIUM *A. Orłowski* (*Fres. Zeits. Anal. Chem.*, XXI, 214) first removes Bi if present, acidifies with  $\text{HCl}$ , decolorizes by  $\text{SnCl}_2$ , adds milk of sulphur and heats to boiling. This precipitates the copper as  $\text{Cu}_2\text{S}$ . From the filtrate the tin is removed by ammonia, leaving the cadmium to be detected in the solution. Another method by the same author is proposed, in which sodium thiosulphate is substituted for  $\text{SnCl}_2$  and milk of sulphur. In other respects it is the same.

The same author (*loc. cit.*) proposes to detect CUPROUS COMPOUNDS IN PRESENCE OF CUPRIC by acidifying with  $\text{HCl}$  and boiling with milk of sulphur. As above, cuprous sulphide will precipitate, while the cupric compounds are unaffected. An elaborate scheme for the EXAMINATION OF COMMERCIAL COPPER for gold, silver, lead, bismuth, antimony, tin, arsenic, iron, cobalt, nickel, zinc, sulphur and phosphorous is given by *R. Fresenius* (*Fres. Zeits. Anal. Chem.*, XXI, 229).

*M. M. P. Muir* (*Jour. Lond. Chem. Soc.*, XLI, 1) determines BISMUTH VOLUMETRICALLY by separating it from other metals, as oxychloride, converting this compound into chloride, dissolving in glacial acetic acid and precipitating by excess of standardized solution of  $\text{K}_2\text{C}_2\text{O}_4$ . The precipitate is  $\text{K}_2\text{C}_2\text{O}_4$ ,  $\text{Bi}_2(\text{C}_2\text{O}_4)_3$ . The excess of oxalate is titrated with permanganate.

For the VOLUMETRIC DETERMINATION OF ANTIMONY *E. T. Herroun* (*Chem. News*, XLV, 101) converts the antimony into  $\text{SbCl}_5$  by treatment with  $\text{HCl}$  and  $\text{KClO}_3$ , boiling gently to expel excess of chlorine, cooling and adding an excess of  $\text{KI}$ . The iodine freed by the action of the  $\text{SbCl}_5$  is then titrated with a standardized solution of sodium hyposulphite.

deposit was obtained after very long standing, but though the test was repeated several times no complete filtration could be effected. Qualitatively the experiment was successful ; the presence of albumen was readily detected in the filtrate on boiling.

As Tolmatscheff\* claims that Hoppe-Seyler's method proved unsuccessful in his hands when applied to milk from women and advises the use of magnesium sulphate to saturation to precipitate casein, and alcohol to precipitate casein and albumen together, experiments were made to test its value in this instance.

By the use of magnesium sulphate the casein was separated, but the milk became so thick and mucilaginous that though left to filter during two days so viscous a mass remained on the filter that all attempts to wash it with magnesium sulphate solution proved futile. The filtrate, what there was of it, was clear and gave a coagulum when heated. The coagulum seemed more copious than that in the filtrates by Hoppe-Seyler's method, but this may have been due to the greater concentration of the liquid.

Not being able to separate the casein by these methods in such form as to weigh it, 20 cc. of the milk were treated by Hoppe-Seyler's method and the albumen determined in 100 cc. of the filtrate. The combined percentages of casein and albumen were then ascertained, which, less the percentage of albumen already found, gave that of the casein. Alcohol proved to be the best precipitant for casein and albumen. The casein separated in fine flocks, carrying albumen and fat with it. The latter was subsequently removed by ether. The liquid filtered nicely, and the precipitate could be thoroughly washed on the filter. The usual method of precipitating albumen and casein by heat and acetic acid gave a precipitate that could not be separated by filtration. Three specimens of milk were collected. Most of the determinations were duplicated. The methods of analysis was the same as that followed in the case of the milk from the elephant Hebe, except as regards albumen and casein. It was not found necessary to dry the milk in vacuo. Once, with the colostrum, sand was mixed with the milk to aid dessication.

The sugar when determined by Fehling's solution volumetrically in the colostrum and No. II. was too low, but estimated gravimetrically in No. III. it agreed closely with the figures in the table. In No. I. the milk treated by alcohol and filtered left on evaporation a residue, less ash, equal to 8.0101 per cent. of sugar.

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\*v. Gorup-Besanez *Physiologische Chemie*, pp. 418 and 420.

	L	II.	III.	IV.
	Feb. 24th.	April 9th.	April 22d.	Milk
	Morning.	Afternoon.	Afternoon.	from Hebe.
Quantity.....	335 cc.	420 cc.	120 cc.	72 cc.
Cream.....	7.7 vol. %	7.7	33	62
Reaction.	Alkaline.	Slightly Acid.	Neutral.	Slightly Acid.
Sp. gr. at 60° F....	1,036	1,036	1,028	1,023.7
In 100 Parts by Weight.				
Water.....	86.5930	82.2390	76.9930	66.697
Solids.....	13.4070	17.7610	23.0070	33.303
Fat.....	3.0100	7.0576	12.6175	22.070
Solids not fat....	10.3970	10.7034	10.3895	11.233
Casein .....	2.0676	5.7060	5.7590	3.212
Albumen.....	0.3455			
Sugar*.....	7.6533	4.5494	4.1510	7.392
Ash.....	0.3306	0.4480	0.4795	0.629
Taste.....	Disagreeable	Much improved.	Excellent.	Excellent.
Odor... ..	Unpleasant	Pleasant	Pleasant	Pleasant
Color.....	{ Yellowish white	White and opaque	White and opaque	White and opaque
Consistency.....	{ Yellow de- posit, other- wise fluid	Very fluid	Fluid	Fluid

Specimen No. III. was drawn after the elephant had been exhibited in the circus circus-ring and while the calf was nursing at the other teat. The calf had not nursed for some hours. Of all it approaches nearest in composition to the milk from Hebe. The solids not fat, remain constant throughout, while the fat varies considerably. Apparatus to serve as a breast pump was constructed in order to collect the milk from one teat or both during say half a day, in order to gain a knowledge of the quantity secreted daily, but the plan unfortunately could not be carried out. “Bridgeport” grew quite as rapidly as the other “baby,” more than doubling her weight during the period of these experiments.

The character of the fat in the last specimen was like that obtained from Hebe, the size and aggregation of the globules and the odor of the fat after extraction being the same. During the last month's of Hebe's period of lactation the employees about the circus frequently partook of her milk and found it most palatable.

\*By difference.



# REVIEW OF PAPERS ON ANALYTICAL CHEMISTRY (IN-ORGANIC) WHICH HAVE APPEARED DURING THE YEAR 1882.

By E. WALLER, PH. D.

A modification of the apparatus for the absolute DETERMINATION OF NITROGEN, invented by S. W. Johnson and E. H. Jenkins, is described by *T. S. Gladding* (*Am. Chem. Jour.*, IV., 42).

According to *A. Guyard* (*Chem. News*, XLV. 159), ALL ACIDS AND OXIDES OF NITROGEN are converted into ammonia by combustion with marsh gas and soda lime. The mixture is made in the proportion of 75 grs. of sodium acetate with  $1\frac{1}{2}$  oz. of soda lime, and the combustion conducted in the ordinary manner. The tube is charged first with about one-quarter of the above mixture, then the remainder mixed with 5 to 7 grms. of the substance to be examined is added, and finally pure soda lime.

The accuracy of the results thus obtained is disputed by *J. Ruffle*, (*Chem. News*, XLV., 186).

In experimenting on methods for the DETERMINATION OF NITROUS OXIDE, *W. Hempel* (*Ber. D. Chem. Ges.*, XV., 903 *et seq.*) finds it extremely difficult to obtain absolutely pure  $N_2O$ . The purest gas obtained contained 98.8%. He finds the best method of estimating the gas to be by explosion with hydrogen in an apparatus specially designed for the purpose, which is carefully described.

100 vols.  $N_2O$  with 460 vols. H was not explosive, while 100  $N_2O$  with 116 vols. H caused such a violent explosion as to shatter the apparatus. The mixture therefore must be made within these limits.

Alkaline solutions of potassium permanganates or of chromic acid are recommended by *C. Boehmer* (*Fres. Zeits. Anal. Chem.*, XXI., 212,) as ABSORBENTS FOR  $N_2O_2$  in analytical processes.

To determine the amounts of sodium SULPHIDE, SULPHITE AND THIOSULPHATE in the same solution, *G. E. Davis* (*Jour. Soc. Chem. Ind.*, I., 89), determines, 1. The amount of iodine absorbed by a measured quantity of the solution, by converting the alkalies present into bicarbonates with carbonic acid water and titrating with iodine solution. 2. The amount of sulphide and thiosulphate present; by adding  $SrCl_2$  in excess, digesting for about half an hour at  $65^\circ$  to  $70^\circ$  C., filtering off the strontium sulphite, washing with air free from water, adding carbonic acid water to the filtrate, and titrating with iodine solution. 3. The amount of thiosulphate



present ; by separating the sulphite as in 2, and then separating the sulphide by digestion for half an hour with  $\text{CdCO}_3$ , filtering and titrating the filtrate with the same precautions as before. A correction must be applied to the results before calculating from the data obtained. It has been found that 100 cc. of a solution of  $\text{SrSO}_3$  saturated at 65 to 70° C., consumes 0.3 cc. decinormal iodine, and the same bulk saturated at 12.7° C. consumes 0.16 cc. decinormal iodine solution. The bulk and temperature of the filtrates must therefore be taken.

To obtain the SULPHUR IN PYRITES in a form for estimation as sulphate, *Fr. Bockmann* (*Fres. Zeits. Anal. Chem.*, XXI. 90), fuses 0.5 gms. with 25 gms. of a mixture containing 6 parts  $\text{Na}_2\text{CO}_3$  to 1 part  $\text{KClO}_3$ .

In estimating CHLORINE GRAVIMETRICALLY, *D. Lindo* (*Chem. News*, XIV., 193) filters the  $\text{Ag Cl}$  through a Gooch filter and dries for half an hour at 140 to 150° C.

In the indirect estimation of CHLORINE AND BROMINE, *L. P. Kinnicutt* (*Am. Chem. Jour.*, IV., 22), after weighing the mixed silver salts in a platinum crucible, reduces to metallic silver by a battery, the crucible being made the positive electrode of the circuit.

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present, are previously converted into ammonia by the action of the zinc-copper couple. The distillate contains all the nitrogen in the form of ammonia.

To convert NITRATES AND NITRITES IN WATER into ammonia, *F. P. Perkins* (*Analyst* VII. 66,) proposes the use of metallic magnesium aided perhaps by the addition of some Na Cl. *J. B. Kinnear* (*Chem. News*, XLVI. 33) finds that zinc with a dilute acid will effect the reduction in about ten minutes, provided that there are about 5,000 parts of liquid present for every part of nitric nitrogen. The rapidity of the reduction depends somewhat upon the amount of surface of zinc exposed.

Among delicate TESTS FOR NITRITES in water, *A. Jorissen* (*Fres. Zeits Anal. Chem.* XXI. 210) proposes a solution containing 0.01 gm. fuchsin in 100cc. of glacial acetic acid, which gives a change of color, passing through blue and green to reddish yellow. (Noted by Vogel *Jour. Prakt. Chem.* XCIV. 457), while *E. W. Davy* (*Chem. News* XLVI. 1) recommends for the same purpose acidifying the solution, adding a few cc. of gallic acid and boiling. The presence of nitrites at once gives a tint to the water similar to that of the Nessler test with ammonia. The reaction is stated to be :



tanno-melanic acid being formed.

The COLOR METER FOR WATERS used by Messrs. *Crookes, Odling, and Tidy* in their examination of the London water (*Report to Local Government Board* for 1881) is described (*Chem. News* XLV. 170). The two-foot tube is used. For comparison colors, two wedges, one brown, the other blue, are used, which are slid over one another until a tint corresponding to that in the water is obtained. The solutions contain respectively :

*Brown*, 0.7 gm. metallic iron as ferric chloride

0.3 " " cobalt as chloride,

and a slight excess of free acid per litre.

*Blue*, 5 gms. pure crystallized cupric acid sulphate per litre. The results are recorded in figures representing the thickness in millimetres of the solutions required to simulate the color of the water thus : Brown : Blue, 20 : 10.

Regarding the examination of fertilizers for REVERTED OR RETROGRADE PHOSPHATES, *C. Richardson* (*Am. Chem. Jour.* IV. 183) finds that the use of ammonium oxalate for their extraction gives variable results depending upon the time and temperature of diges-

tion, the degree of concentration of the solution, &c. In this connection see also results of *T. S. Gladding* (this *Journal*, p 113). *F. J. Lloyd* (*Jour. Lond. Chem. Soc.* XLI. 306) triturates 0.5 gm. of the fertilizer with 20cc. of a solution containing 30 per cent. citric acid, and enough ammonia to render it strongly alkaline. The mixture is made up to 100cc. and allowed to stand over night at the temperature of the laboratory. In 50cc. of the clear solution the lime is separated by acidifying with acetic acid and adding ammonium oxalate, and in the filtrate the phosphoric acid is determined by the use of magnesia mixture.

As to VOLUMETRIC processes for PHOSPHORIC ACID, *C. Mohr* (*Fres. Zeits. Anal. Chem.* XXI. 216) shows by a series of experiments that even small amounts of ammonia salts have a marked effect on the results obtained by the uranium titration, while *H. Pemberton* (*Jour. Franklin Inst.* CXIII. 184). proposes the use of a solution of neutral ammonium molybdate containing 89.543 gms. of the salt per litre (1cc=0.003 gm.  $P_2O_5$ ). For titration 100 to 125cc. of the solution is taken [which should not contain over 0.1 gm.  $P_2O_5$ ] 10 gms of ammonium nitrate, and 2cc. of nitric acid [sp. Gr. 1.14] are added and the solution is heated to 140° F. or over. The end reaction is the absence of further precipitation. Toward the close of the titration portions of the solution have to be filtered to give opportunity to observe the end reaction. If the titration is overrun, a measured quantity of  $Na_2HPO_4$  solution of known strength is added and the titration resumed. From the burette reading 0.5cc. is to be deducted for the effect of the nitric acid added. Silica and organic matter cause erroneous results when present, while chlorides, iron [to the extent of 0.1 to 0.3 gm.] &c., do not.

In the EXAMINATION OF TITANIFEROUS IRON ORES, *T. M. Drown and P. W. Shimer* (*Am. Chem. Jour.* IV. 1), treat the ore (2 to 5 grms.) with 50cc. of H Cl. (Sp. Gr. 1.12) evaporate to dryness and heat in the air bath for an hour, add 50cc more H Cl., heat and filter; turbidity in the washings is prevented by the use of ammonium nitrate. The phosphoric acid is extracted from the residue by fusion with sodium carbonate, extracting with water, evaporating to dryness with nitric acid to remove silica, &c. Molybdate mixture may then be added to the solution. In the first solution (obtained by H Cl.) the H Cl. is removed by evaporation with nitric acid, when after partially neutralizing, molybdate solution is added. This last molybdate precipitate requires purifying by dissolving in ammonia, treating the undissolved residue with nitric acid and pre-

precipitating that solution with more molybdate. After obtaining all the ammoniacal solutions of the phospho molybdate free from silica, titanio acid, &c., they are united and precipitated as usual with magnesia mixture.

For TITANIO ACID these gentlemen decompose the ore by fusion with  $KHSO_4$  and after neutralizing and reducing with  $SO_2$  precipitate by boiling as usual. *P. T. Austen* and *F. A. Wilber* (*Am. Chem. Jour.* IV, 211) find that the addition of 50 to 60cc. of acetic acid aid materially in the precipitation of the titanio acid, giving good results on boiling, even when chlorides are present. The last named decompose the ore by fusion with alkaline carbonates and treating with  $HCl$ , evaporating, &c.

For the *determination* of CARBON AND SILICON IN IRON, STEEL, etc., *F. Watts* (*Chem. News*, XLV., 279) gives the preference to a modification of Wohler's method.—Volatilization of the iron in a stream of  $Cl$  gas and making a combustion of the carbon remaining. Two operations are required for each analysis. In the first after volatilizing the iron in a current of *dry* chlorine free from oxygen, the residue is submitted to combustion, and the *total carbon* is thus determined. In the second the residue (after volatilizing iron) is weighed, giving *total carbon and slag*, while the gases from this operation are passed into water, where the  $SiCl_4$  is decomposed, and by boiling, evaporating to dryness, etc., the  $SiO_2$  corresponding to *combined* silicon is obtained.

SODA COMPOUNDS may be estimated in potassium carbonate, according to *A. Van Husselt* (*Jour. Soc. Chem. Ind.* I, 203), by treating a weighed quantity (converted into chloride) with  $HCl$ . (Sp. Gr. 1.189) saturated with  $NaCl$ .

To estimate CAUSTIC ALKALI IN PRESENCE OF CARBONATE, *R. B. Warder*, (*Sci. Proc. Ohio Mech. Inst.* I, 45) uses a process based upon the fact that phenolphthalein is colorless in the presence of alkaline bicarbonates. On titrating a very dilute solution of the sample (cold) until the phenolphthalein which has been added imparts no color to the solution, all the caustic and half of the carbonated alkali is neutralized, giving a result A. Then by boiling and titrating the remainder of the alkali is neutralized; result B.  $A+B$  measures total alkalinity, and  $A-B$  measures caustic alkali.

To detect and separate ALKALIES IN ARGENTIC NITRATE. *M. Stolba* (*Jour. de Pharm. et de Chim.*) uses hydrofluosilicic acid. By the use of phenolphthalein *M. Richter* (*Fres Zeits. Anal. Chem.*, XXI, 205) determines acidimetrically the CHROMIC ACID IN

POTASSIC DICHROMATE. Many other indicators cannot be used on account of the oxidizing action of the chromic acid upon them.

By fusing hydrorufigallic acid with potassa a NEW INDICATOR for alkalimetric titration is made by *J. Oser and W. Kalmann* (*Monats hefte f. Chemie*, II, 50). It dissolves in alkalies with an olivegreen tint—a slight excess of alkali gives carmine red, changed to yellow by free mineral acids. It is unaffected by  $\text{CO}_2$ .

To DETERMINE ZINC in its ores, *A. Millot* (*Bull. Soc. Chim.*, XXXVII, 339) recommends dissolving with  $\text{HCl}$  and  $\text{KClO}_3$ , evaporating, adding ammonia and ammonium carbonate, diluting the filtrate therefrom, and in an aliquot portion of this filtrate, adding a little  $\text{K Cy}$  and precipitating by the battery. The addition of ammonium acetate prevents the corrosion of the electrodes.

In TITRATING ZINC by Fahlberg's method manganese must be first separated to obtain correct results. *R. W. Mahon* (*Am. Chem. Jour.*, IV, 53). In titrating with  $\text{Na}_2\text{S}$ , the use of paper soaked in thallium solution is recommended by *M. Schröder* (*Berg. u. Huttenmann, Zeit.* 1882, p. 4) as an indicator. If a slight excess of ammonia is present the thallium salt is unaffected by  $\text{Zn S}$ .

To separate NICKEL FROM COBALT *G. Delvaux* (*Comptes Rendus*) dissolves in aqua regia with excess of  $\text{HCl}$ —adds ammonia in excess, then potassium permanganate until the rose color holds for some time. Solution of pure  $\text{KHO}$  will then precipitate nickel (with manganese if present), leaving cobalt in solution. In case small amounts of nickel and large amounts of cobalt are present *A. Jorissen* (*Fres. Zeits. Anal. Chem.*, XXI, 208) precipitates with  $\text{NaHO}$ , adds bromine, then a few cc. of  $\text{K Cy}$ , and agitates in the cold. The cobalt is dissolved while the nickel remains in the precipitate.

*A. Carnot* (*Bull. Soc. Chim.*, XXXVII, 482) ESTIMATES CHROMIUM (as base) by adding sodium acetate to the solution slightly acidified, adding an alkaline phosphate and boiling. The presence of oxalates must be avoided. The precipitate dried at  $100^\circ$  contains  $6\text{H}_2\text{O}$ . Ignited it consists of  $\text{Cr}_2(\text{PO}_4)_2$ , containing 51.86 per cent.  $\text{Cr}_2\text{O}_3$ .

In the examination of iron ores, etc., containing chromium error may be caused, according to *B. Reinitzer* (*Monats hefte f. Chem.*, 1882, p. 249), by the chromium preventing the precipitation of some of the iron and alumina on boiling the solution after neutralizing and adding sodium acetate.



For the detection of IMPURITIES IN COPPER several chemists communicate their methods—

For ARSENIC *J. Pattinson* (*Newcastle-on-Tyne Chem. Soc.*, Feb. 1882) dissolves in nitric acid; then by adding sufficient soda solution to a little more than neutralize the free acid present, the arsenic is precipitated as copper arsenate; after standing for half an hour with frequent stirring the precipitate is filtered and washed cold. The arsenic is thus concentrated in the precipitate. *A. H. Sexton* (*Chem. News*, XLV, 255) prefers to precipitate the arsenic from the nitric acid solution (by adding ferric nitrate, neutralizing, adding sodium acetate and boiling) as basic acetate. The precipitate is dissolved in acid, precipitated by  $\text{H}_2\text{S}$ , oxidized to arsenic acid, and determined as the magnesia compound.

For CADMIUM *A. Orłowski* (*Fres. Zeits. Anal. Chem.*, XXI, 214) first removes Bi if present, acidifies with  $\text{HCl}$ , decolorizes by  $\text{SnCl}_2$ , adds milk of sulphur and heats to boiling. This precipitates the copper as  $\text{Cu}_2\text{S}$ . From the filtrate the tin is removed by ammonia, leaving the cadmium to be detected in the solution. Another method by the same author is proposed, in which sodium thiosulphate is substituted for  $\text{Sn Cl}_2$  and milk of sulphur. In other respects it is the same.

The same author (*loc. cit.*) proposes to detect CUPROUS COMPOUNDS IN PRESENCE OF CUPRIC by acidifying with  $\text{HCl}$  and boiling with milk of sulphur. As above, cuprous sulphide will precipitate, while the cupric compounds are unaffected. An elaborate scheme for the EXAMINATION OF COMMERCIAL COPPER for gold, silver, lead, bismuth, antimony, tin, arsenic, iron, cobalt, nickel, zinc, sulphur and phosphorous is given by *R. Fresenius* (*Fres. Zeits. Anal. Chem.*, XXI, 229).

*M. M. P. Muir* (*Jour. Lond. Chem. Soc.*, XLI, 1) determines BISMUTH VOLUMETRICALLY by separating it from other metals, as oxychloride, converting this compound into chloride, dissolving in glacial acetic acid and precipitating by excess of standardized solution of  $\text{K}_2\text{C}_2\text{O}_4$ . The precipitate is  $\text{K}_2\text{C}_2\text{O}_4$ ,  $\text{Bi}_2(\text{C}_2\text{O}_4)_3$ . The excess of oxalate is titrated with permanganate.

For the VOLUMETRIC DETERMINATION OF ANTIMONY *E. T. Herroun* (*Chem. News*, XLV, 101) converts the antimony into  $\text{Sb Cl}_5$  by treatment with  $\text{HCl}$  and  $\text{KClO}_3$ , boiling gently to expel excess of chlorine, cooling and adding an excess of  $\text{KI}$ . The iodine freed by the action of the  $\text{Sb Cl}_5$  is then titrated with a standardized solution of sodium hyposulphite.

*M. M. P. Muir* (*Chem. News*, XLV, 69) detects TIN IN THE PRESENCE OF ANTIMONY by boiling the HCl solution for 10 minutes with copper turnings which reduces it to stannous form and permits of its being detected by HgCl<sub>2</sub>.

To detect SMALL AMOUNTS OF SILVER IN LEAD (reduced from galena, etc.) *J. Krutwig* (*Ber. D. Chem. Ges.* xv, 307) dissolves in nitric acid and precipitates with large excess of NaHO. Silver plumbate remains insoluble. By extracting with ammonia, evaporating to dryness, dissolving in nitric acid and separating the lead by H<sub>2</sub>SO<sub>4</sub>, a solution is obtained in which the silver may be detected with HCl.

TO SEPARATE GALLIUM from zinc and ferrous oxide *L. de Boisbaudran* (*Comptes Rendus*, 1882, No. 17) uses cupric hydrate instead of barium or calcium carbonate. The copper may afterward be separated by dissolving and adding H<sub>2</sub>S.

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## ABSTRACTS.

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Abstracts from *Journal of the Chemical Society*, London, by Arthur H. Elliott, Ph. B., F. C. S.

**On some Higher Oxides of Manganese and their Hydrates.** (Part II., Vol. XL., p. 56). BY V. H. VELEY, B. A., F. I. C.

The higher oxides of manganese, when heated in dry nitrogen at temperatures ranging from 60°—200°C., are simply dehydrated without loss of available oxygen; the dehydrated oxide formed readily absorbs oxygen. The oxides, when heated in dry hydrogen, are simultaneously dehydrated and reduced; a hydrate of the red oxide is formed which readily absorbs oxygen. The quantity of water retained by the peroxides when heated to 200°C. in dry hydrogen, is greater than that retained by the same oxide at the same heat in air, oxygen, or nitrogen.

**On a New Alkaloid from Cinchona Bark.** (Vol. XL., p. 66) BY DAVID HOWARD AND JOHN HODGKIN.

The alkaloid is obtained from *China Cuprea* of Dr. Fluckiger. Its specific rotary power is very near that of quinine: 100 cc. of ether free from alcohol dissolves 0.57 of it at 12°C. Ether containing 8 per cent. of alcohol dissolves 2.27. Alcohol of 90 per cent. at 12°C. dissolves 7.64 per 100 cc. When impure or mixed with



other alkaloids it is much more soluble. Authors describe the sulphate, bisulphate, oxalate, tartrate, hydrochloride, and the iodosulphate ; also the platinum salt and the action of chlorine and ammonia.

**Contributions to the Chemistry of Rare Earth-Metals.** (Vol. XL., p. 68). BY B. BRAUNER, PH. D.

The object of this research is the study of the cerite metals, with special reference to their position in the periodic system. The metals cerium, lanthanum, and didymium, are each found to occupy its own characteristic position in the system.

**Note on Certain Photographs of the Ultra-violet Spectra of Elementary Bodies.** By W. N. HARTLEY, F. R. S. E. Vol. XL., p. 84. An account of the application of the new dry plate process of photography to the production of spectra-photographs. By means of prisms and lenses of quartz or Iceland spar, photographs were obtained which contained the whole of the lines of metallic spectra completely in focus on one plate, and extending from the blue to the extreme ultra-violet rays. By prolonged exposure of gelatin plates photographs to the red end of the spectrum may be obtained. The paper is illustrated with Woodbury-types of the spectra.

**The Chemistry of Bast Fibres.** By C. F. CROSS and E. J. BEVAN. Vol. XL, p. 90. Authors give an account of their researches upon the action of halogens upon jute and their fibres; more especially the action of bromine. Also the action of dilute sulphuric acid upon fibres. They are led to conclude that jute fibre is composed for the most part *not* of cellulose, but of a transition form of cellulose from that of the original carbohydrate to its ultimate modification of a soluble astringent. They call the transition modification of cellulose *Bastose*, and give the name *Bastin* to the aromatic derivatives of cellulose. Incidentally they discuss Glycolignose and Glycodrupose; the origin of tannins; reactions of the jute substance under high pressure; reduction of indigo by jute; and reaction of linseed oil with sulphuric acid. In conclusion they give a diagram showing the passage of cellulose into aromatic bodies allied to the tannins.

**A New Apparatus for the Determination of Melting Points.** By C. F. CROSS and E. J. BEVAN. Vol. XL., p. 111. A small cup is attached to the bulb of a thermometer, and in this cup

some of the body to be tested is melted. While the body is still liquid a minute bulb tube, with a piece of platinum wire fixed in at the bottom, is dipped into the liquid and allowed to stay there till the whole is solidified. To determine a melting point the combination is placed in mercury, the latter is heated, and when the body melts the bulb tube rises to the surface and the thermometer registers the melting point at that instant. Authors state that no stirring is necessary during the heating of the mercury.

**On the Reaction of Chromic Anhydride with Sulphuric Acid.** By C. F. CROSS and A. HIGGIN. Vol. XL., p. 113. Authors examined the insoluble bodies formed by the action of sulphuric acid upon chromic anhydride. They find they are only approximately constant in composition, and give as an average formula  $4 \text{Cr}_2 \text{O}_3 \cdot 7 \text{H}_2\text{O} \cdot 13 \text{SO}_3$ , or  $2 (2 \text{Cr}_2 \text{O}_3 \cdot 3 \text{SO}_3) \cdot 7 \text{H}_2\text{SO}_4$ . These bodies are entirely insoluble in water, and are attacked with difficulty by solutions of alkaline hydrates. Potassium dichromate and sulphuric acid give coarsely granular precipitates that contain potassium.

**On Some Decompositions Produced by the Action of Chloride of Aluminium.** By C. FRIEDEL and J. M. CRAFTS. Vol. XL., p. 115. Paper contains remarks upon E. H. Rennie's note (p. 33) on the action of ethyl chlorocarbonate on benzene. Authors state that they had attained exactly the same results, and described them in *Compt. rend.*, June 18th, 1877. They are now engaged in the study of the destructive action of aluminium chloride upon hydrocarbons, and state that the higher homologues of benzene lose their side groups and tend to be resolved into benzene when heated with aluminium chloride. Hexmethyl-benzene is easily reduced to durene and simpler hydrocarbons.

**Chemical Examination of the Buxton Thermal Water** By J. C. THRESH, B. Sc., Vol. XL., p. 117. The density of the water at  $25^\circ.8 \text{ C.}$  was 0.99686; and when compared with pure water at the same temperature was 0.99992. The author gives minute details of his methods of analysis in which he follows the course of procedure recommended by Bunsen. The paper contains elaborate tables showing the analyses of the portions of the total residue both soluble and insoluble in water.

For comparison with the results of other analysts the author gives his results in the following form; in which the constituents are arranged as parts in 10,000.

Temperature of water.....81.7° F  
 Specific gravity.....0.99992  
 Total solids.....2.934

Ca 0.567—Ba 0.004—Sr trace Mg 0.195—Na 0.213—K 0.040—  
 Li trace—NH<sub>4</sub> 0.0001—Al trace—Fe 0.002—Mn 0.0014—Pb trace—  
 CO<sub>2</sub> 1.238—F 0.0014—Cl 0.370—I nil—SO<sub>4</sub> 0.160—NO<sub>3</sub> 0.003—  
 Organic matter 0.003—Si O<sub>2</sub> 0.136—PO<sub>4</sub> trace.

And the following elements were detected in the sinter Mo, Cu, Co and Zn.

The gaseous constituents in cubic centimetres per litre at 0° C and 760 m. m. pressure were CO<sub>2</sub> 14.56—N 22.13.

The ammonia was determined by distilling with sodium carbonate and estimating with Nessler's solution in the distillate. The nitric acid was obtained by digesting with zinc-iron couples and then distilling and estimating the resulting ammonia by Nessler's solution. Nitrous acid was sought but not found.

**Dibenzoylaniline and its Isomerides.** By A. Higgin, Vol. XL., p. 132. The author has repeated the work of Gerhardt in obtaining dibenzoylaniline by the action of benzoyl chloride upon monobenzylaniline; also the work of Losanitch in obtaining phenyldibenzoylamide by the action of benzoic acid upon phenyl thiocarbimide at 130–150° C. The author states that it is necessary not to use the thiocarbimide in excess. Phenylbenzoyl-benzoylamide was prepared by acting upon phenylbenzoylamide with benzoyl chloride in presence of zinc chloride. It crystalizes in colorless plates melting at 150°; and when decomposed with dilute hydrochloric acid yields benzoic acid and a solid base, benzoyl-phenylamine. The sulphate of this base was made, also the chloride and a platinochloride. In the alcoholic solution from which the phenylbenzoyl-benzoylamide had crystallized an isomer of this body was obtained.

**Contributions to Our Knowledge of the Composition of Alloys and Metal Work, for the Most Part Ancient.** By WALTER FLIGHT, D. Sc., F. G. S. Vol. XL., p. 134.

Author gives a number of facts relating to the history of copper-nickel coinage. Coins of Ancient India 500 B. C. contained Ag 89.120—Ag Cl 1.328—Au 1.253—Cu 4.337—Pb 3.735—Graphite 0.075. A figure of Buddha contained Ag 57.701—Ag Cl 4.113—Cu 37.548—Au 0.483—Graphite 0.403. Bidrai Ware contained Zn 94.552 to 93.516—Cu 3.920 to 3.278—Pb 1.400 to 2.171

—Au nil to 0.690—Fe 0.128 to 0.345. A sickle found by Belzoni under the feet of a sphinx at Karnak, Egypt, contained: Silica 11.884—Magnetite 5.111—Calcium carbonate 0.172—Ferric oxide 64.604—Water 18.229—Nickel trace. A double hook of "Bronze" found in one of "Dixon's Passages" of the Great Pyramid contained Cu 99.521—Fe 0.479. "Bronze" figures from Egypt contained Cu 68.421—Fe 4.694—Pb 22.759—Ni (Co) 0.782—Sn 0.938—As 1.479—Sb 0.668. Another "Bronze" figure from Egypt contained Cu 82.19—Pb 15.79—Sn 2.02. Three copper spear heads from Cyprus contained :

	I.	II.	III.
Cu.....	97.226	98.398	99.470
Fe.....	1.322	0.729	0.384
Ni.....	—	0.153	0.084
Au.....	0.279	0.305	—
Pb.....	0.076	—	—
Sn.....	trace	—	—
As ..	1.348	trace	trace
S.....	—	0.305	—

All contain traces of P.

Copper axe-heads from Beth Saour, near Bethlehem, Holy Land, contained 99.557 and 99.93 per cent. of copper, with traces of As, Fe, Ni and S. Hebrew shekel contained Ag 97.674—Au 0.676—Cu 0.642—Graphite 0.034. The following bronzes : I, sword or dagger blade from Cyprus ; II, Part of figure of Mercury, of Romano-British times, from Leadenhall street, London ; III, Handle of vase, found at Island of Tilos, of about the time of Alexander the Great ; IV, Roman bronze figure in the Blacas Collection of British Museum, had the subjoined composition :

	I.	II.	III.	IV.
Cu.....	88.771	78.355	81.764	87.158
Sn.....	8.508	10.018	10.901	10.724
Pb.....	1.504	9.302	5.246	2.005
Fe.....	0.476	0.715	0.153	trace
Co.....	0.304	—	1.222	—

Ni, P, Au and Ag were only found in traces.

An Incas Pin, found on a mummy at Arica, S. Peru, after earthquake 1868, contained Ag 82.222—Ag Cl 1.388—Au 0.406—Cu 16.100—Fe 0.119. Bronze bar, from ruins of Temple in Bolivia contained Cu 93.26—Sn 6.52—Fe 0.26—Ni and As traces. These

results are given with a great many interesting details as to the origin and antiquity of the various objects examined.

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Abstracts from *Berichte der Deutschen Chemischen Gesellschaft*, by L. H. Friedburg.

**Relations between the amounts of rotation on the plane of polarization, F. Flawitzky** (Vol. XV., p. 5).

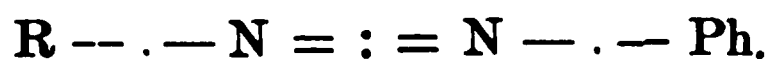
Critique of Krecke's first law: "An optically active substance, forming a combination with an optically non-active one, or modified by chemical agents, either maintains its molecular power of rotation unchanged, or the latter is a simple multiple of the former." The author finds that under certain conditions and regarding substances genetically related, the relations between the molecular power of rotation are independent of concentration. Such an independence might indicate eventually the immutability of optical activity in chemical transformations.

**The Molecular Power of Refraction of the Terpenes. F. FLAWITZKY.** (Vol. XV., p. 15).—The difference of molecular refraction of the Terpenes correspond to their atomicity.

**On the Action of the two Isomeric Mono-Brom-Cinnamic Acids with Conc. Sulphuric Acid. R. LEUCKART.** (Vol. XV., p. 16).—A remarkable difference is manifested in treating either Alpha or Beta mono brom-cinnamic acid with conc. sulphuric acid, which acts as an agent of condensation. Pure Alpha acid is hardly attacked, if at all, whilst the isomeric Beta acid is decomposed, though the products of decomposition and the constitution of these products are not yet either fully studied or known.

A constitutional similarity between these acids and fumaric and maleic acid seems possible.

**Azodyestuffs. O. WALLACH.** (Vol. XV., p. 22).—Diazo compounds and phenols have of late been combined to a large series of dyestuffs. The general formula of these might be expressed thus:



R meaning any monovalent radical of an aromatic compound (thus:  $\text{C}_6\text{H}_5 \text{---} . \text{---}$ ;  $\text{C}_6\text{H}_4\text{SO}_3\text{H} \text{---} . \text{---}$ ;  $\text{C}_{10}\text{H}_7 \text{---} . \text{---}$  &c.), and Ph understood as any monatomic rest of a phenol (e. g.:  $\text{C}_6\text{H}_4\text{OH} \text{---} . \text{---}$ ;  $\text{C}_6\text{H}_3(\text{OH})_2 \text{---} . \text{---}$ ;  $\text{C}_{10}\text{H}_6(\text{SO}_3\text{H})(\text{OH}) \text{---} . \text{---}$  &c.).

Combinations with more than one azo group have been prepared only sporadically, and such with *different* azo-groups in *one* rest of phenol only once by Griess.

Wallach prepares combinations of diatomic phenols with two similar or dissimilar diazo-rests.

General formula:



In which R and R<sup>1</sup> may be identical or different.

Further on the question is put, whether it makes any difference, or not, in what consecutive order *different* azo-rests are incorporated into a diatomic phenol.

In pursuing those questions, the author obtains a series of new dyestuffs, the preparation of which has been patented.

**On Caffein.** EMIL FISCHER. (Vol. XV., p. 29).—The author continues experiments to prove the constitutional formula of his synthetic caffein, and consecutively he gives such fourmula for caffolin, hydroxy-caffein, apocaffein, hypocaffein. Finally his endeavors have resulted in producing a caffuric and hydrocaffuric acids, and theobromin.

**The Action of Chloride of Aluminium on Mono-Brombenzol.** O. V. DUMREICHER. (Vol. XV., p. 33).

**On Gum Arabic.**—HEINRICH KILIANI, (Vol. XV. p. 34). A comparison of ten different samples of gum arabic, (including two samples of arabic acid) in regard to rotatory power and percentage of mucic acid.

**Occurrence of large Crystals of Ammonium Magnesium Phosphate in Urine Nearly 100 Years Old.** H. SCHWANERT. (Vol. XV. p. 37). A bottle with urine was found some time ago in a coffin along with a perfectly preserved human skeleton. The coffin was found underneath a street at Wadersloh, in Westphalia by Dr. Gruwe. The street bordered an old cemetery, not used since 1830 as burial ground. In 1827 or 1828 this street was built on part of the cemetery. The bottle with urine, a green, short-necked cylindrical bottle, stoppered with a dark-brown colored cork, pressed somewhat into the neck, had most likely been put into the coffin from superstitious motives. Such superstition exists even to day in that part of the country.

The sediment of the urine consisted of microscopical white and

brown particles, the former being carbonate of calcium the latter urate of ammonium showing the murexide reaction. The three larger crystals, one of which was 8 mm. long were ammonium magnesium phosphate.

**Expulsion of the Sulpho-Group by Bromine.** WERNER KELBE. (Vol. XV. p. 39). Sulphurous acid, bromine and water, yield hydrobromic and sulphuric-acids.

An aromatic sulpho-acid, (which can be regarded as a substituted sulphurous acid) bromine and water, yield at a certain temperature hydrobromic and sulphuric acids, and a monobrominated hydrocarbon.

Kelbe thus prepared: Alpha—Brommeta-iso-cymol and Beta—Brommeta-iso-cymol.

**Reaction of Phosgen on Diazo-Amido Compounds.** A. SARAUW, (Vol. XV. p. 42). This research shows, that neighboring positions must be ascribed to: the imido-group and the tolyl-rest in diazo-benzol-para-toluidide; the imido group and the carboxyl benzol-rest in diazo—amido—benzoic acid; the imido-group and the bromphenyl-rest in diazo-benzol-parabrom-anilid. Therefore it is likely, that in the mixed diazo-amido-compounds, the imido group is combined with the more negative aromatic rest.

**On Tetrabrombenzol.** RICHARD MEYER, (Vol. XV. p. 46)., Bromine and nitrobenzol brought together at a high temperatures from the *same* tetra-brombenzol as is generated by the reaction between para-di-brombenzol and an excess of bromine at + 150°C.

Hence Kekule was right, in supposing that his combination, in spite of great differences in the data of Riche and Bérard, was the same, which these gentlemen prepared by means of paradibrombenzol.

**Halogenized and Hydroxylized Organic Acids.** E. ERLÉN MEYER and C. L. MULLER (Vol. XV. p. 49).

**Combination in the Indigo Group.** ADOLF BAEYER (Vol. XV. p. 50). The intermediate products between ortho-nitro-phenyl-propionic acid and indigo are here studied by the author because he thinks, that the formation of indigo out of the first named product is due to an intermediate product formed, and by oxydation transformed into indigo.

Consequently ortho-dinitro-diphenyl-diacetylen was prepared, which, treated with fuming sulphuric acid gives rise to diisatogen,

a substance of remarkable interest. Of all artificially prepared substances it stands nearest to indigo and is easiest transformed into indigo; ammoniac sulphhydrate transforms it, cold, quantitatively into indigo.



The color is formed directly, without preliminary formation of indigo white or indoxyl, the diisatogen in contact with reducing agents becoming blue without either being dissolved or changing in form.

**Syntheses by Means of Phenyl-Acetylene and its Derivatives.** ADOLF BAEYER and LUDWIG LANDSBERG (Vol. XV. p. 57). Preparation of diphenyl-diacetylen, and other derivatives.

**Nicotinic Acid from Pyridin.** OTTO FISCHER. (Vol. XV., p. 62.) Pyridin-sulpho-acid was transformed into cyanpyridin, which, treated with conc. hydrochloric acid, gave pyridin-monocarbonic acid, which latter is identical with nicotinic acid. A comparison of this acid and the nicotinic acid prepared from chinolin showed no difference whatever in properties.

**Phoron from Glycerin.** K. E. SCHULZE. (Vol. XV., p. 64.) Production of phoron from glycerin by fermentation.

**Refraction and Heat of Combustion.** JULIUS THOMSEN. (Vol. XV., p. 66.)

**Normal Cyanic Acid and Derivatives.** E. MULDER. (Vol. XV., p. 69.) Preparation of ethyl ether of cyanic acid.

**The Reducing Properties of Living Cells.** J. REINKE. (Vol. XV., p. 107.) Controversial.

**Contributions to the Chemistry of the Cerium Metals.** B. BRAUNER. (Vol. XV., p. 109.) Studies in regard to the periodical system of the elements.

**Synthesis of the so-called Alkyldisulphoxides.** ROBERT OTTO. (Vol. XV., p. 121.)

**Contributions to the History of Atropin.** A. LADENBURG. (Vol. XV., p. 133.) Controversial.

**On Determination of the Density of Gases.** HEINRICH GOLDSCHMIDT and VICTOR MEYER. (Vol. XV., p. 137.) Description of apparatus, &c.



## ON THE ESTIMATION OF SULPHURETTED HYDROGEN IN GAS BY DIRECT WEIGHT.

BY A. P. HALLOCK.

Last winter, while engaged in making some investigations in water gas, I made a few experiments in regard to the sulphur present, in order to see if I could not obtain some idea of in what form it was present. To do this I started to make some determinations of the total amount of sulphur in the gas. Using first the finished gas, which is the gas as it goes to the consumer, after being both enriched and purified, next the gas which had only been enriched but not purified, and lastly what is called the hydrogen. This is the water gas just as it comes from the gasogens, and before the addition of the naphtha vapor. It consists of a mixture of carbonic oxide, hydrogen, marsh gas, carbonic acid and sulphur compounds in varying proportions depending upon the kind of coal used.

With the finished gas I had but little trouble, for the total sulphur could be estimated in the same way as the sulphur in coal gas, by burning it in what is known as the Referee's apparatus. In this apparatus, the gas is burned very slowly at the lower end or mouth of a vertical trumpet-shaped chimney; the upper end of the chimney having the small opening is connected with a condensing cylinder filled with glass beads in order to present the largest possible amount of surface. On a collar around the burner are placed some pieces of solid carbonate of ammonia, or a dish containing strong ammoniac hydrate may be placed under the burner. Ammonia vapor is drawn up through the chimney by the current of air which supports combustion, and unites with the sulphurous acid to form a salt which condenses in the cylinder. After a little while the liquid condensed in the cylinder begins to trickle down the glass beads to the bottom, where it runs out through a small tube into a beaker or flask set underneath. After ten or more feet of gas have been burnt, the gas is turned off, the cylinder and beads washed, and the washings added to the contents of the beaker. After evaporating down to small bulk, the sulphur compounds are oxydized with bromine or chlorine to sulphuric acid, combined with barium, weighed as sulphate of barium, and from this the sulphur calculated. If the oxydation has been complete, the liquid dropping into the beaker will be colorless

or very nearly so, but when the sulphur has not been completely oxydized the liquid will have a decidedly yellow color.

With the finished gas the solution was always colorless or very nearly so, but when I attempted to estimate the sulphur in the unpurified gas, either before or after enriching, I always obtained a deeply-colored liquid, and the total amount of sulphur but little more than the amount obtained from the purified gas, and much less than I had reason to suppose existed. Why this should be, I am as yet unable to explain, but as it was actually the case, after a few repeated attempts I gave it up and started to make estimations of the amount of sulphuretted hydrogen, thinking if I could accomplish this it might give me some idea of the sulphur.

I tried making some determinations in an absorption tube for gas analysis, using binocide of manganese as recommended by Prof. Bunsen, but I soon found that in cases like this where the quantity of sulphur was comparatively small and the amount of gas which could be used was also very small, the liability to error was so great that no satisfactory results could be obtained. I then concluded to try it by absorption in a U-tube in order to employ a large volume of gas. At first liquids were used, such as solution of acetate of lead, sulphate of copper, etc., but none of these worked well. At the suggestion of Mr. Lees, I tried dehydrated sulphate of copper, this absorbed the sulphuretted hydrogen very nicely, forming sulphide of copper and sulphuric acid; but the difficulty was that in order to completely dehydrate it, it was necessary to pulverize it, and in so doing, it generally became so finely powdered that it would pack in the U-tube and clog it up until the pressure would become sufficient to blow up through it, forming direct gas passages, through which the gas would pass and hardly be acted upon. About this time I was busily engaged with some other work and had no more time to devote to the sulphur determinations and left them for awhile. In the meantime, which was about the first of April, Mr. Lewis T. Wright, of England, published an article in the English *Gas Light Journal* on the estimation of sulphuretted hydrogen and carbonic acid in coal gas. In this article he mentioned the difficulty offered to the passage of the gas in the U-tube by the ordinary dehydrated sulphate of copper, and said he had overcome this by drying it first at about  $100^{\circ}\text{C}$ ., then adding a little water to it and stirring it with a glass rod in order to slake it, which makes it granular, then drying it again at  $100^{\circ}\text{C}$ . and then at  $155^{\circ}\text{C}$ . At my first opportunity I prepared some in this manner and found it porous, and

it worked very nicely, completely overcoming the previous difficulty.

In order to give a better idea of the method employed, I will describe the apparatus which was used. It consisted first of a drying cylinder containing fused chloride of calcium, next to this was attached a weighed six-inch glass stoppered U tube containing the dehydrated sulphate of copper, a bit of cotton was put on the top of the sulphate in the U-tube to prevent any of it being blown out. To this U-tube was attached another weighed U-tube containing soda-lime and chloride of calcium to catch any carbonic acid and thereby enable me to determine it at the same time, and after this was placed, a meter to measure the quantity of gas passed through the tubes, and from the meter was a tube leading to a small gas burner where the gas was burnt.

The object of the first chloride of calcium was to dry the gas, for any moisture would be caught in the sulphate of copper tube and weighed as sulphuretted hydrogen. In burning the gas in the Referee's apparatus, I had to pass it through a wet meter first, to measure it, and in order to avoid a loss by the sulphur being washed out by the water, the water was replaced by glycerine and gas allowed to pass through it for a long time before using it, but in this case I was able to place the meter after my absorption apparatus, and thus avoid any error from this cause.

The apparatus was first tried on the pipe leading from the hydrogen main; after letting a sufficient quantity blow out to ensure that it was fresh gas, for when the gas was first turned on it failed to produce any stain on acetate of lead paper showing that the sulphur was being removed from the gas by the iron rust in the pipe. Sometime after it began to produce a stain, the apparatus was attached by means of a T-shaped pipe so that the gas could be kept burning all the time and thus have the flow of gas up the iron pipe much more rapid, and thereby have it as little acted upon as possible.

The gas was allowed to pass very slowly through the apparatus, not over one foot in three hours; after one foot had passed, the U-tube was detached and weighed, and found to have increased 0.198 grammes in weight, which would give 315.51 grains of sulphuretted hydrogen in 100 cubic feet, or 0.47 per cent. by volume. After this, four more determinations were made, allowing only one-half foot to pass each time, and although in this case the results had to be multiplied by 200 they agreed very closely, as may be seen by the following table. The column marked A gives the increase

in grammes of weight of the U-tube, B gives the number of grains of sulphuretted hydrogen in 100 cubic feet, and C the percentage :

	A.	B.	C.
No. 1.....	.1085	344.91	0.51 per cent.
No. 2.....	.1100	339.52	0.52 “ “
No. 3.....	.1110	342.59	0.52 “ “
No. 4.....	.1105	341.00	0.52 “ “

These I consider as agreeing very closely.

A few days after I began the experiments upon the finished gas and was surprised to find that the U-tube gained more in weight than it did when using the unpurified, sometimes showing as much as 100 grains more in 100 cubic feet, while the Referee's apparatus, as used by Dr. Love and also by myself, did not give over 10 grains of total sulphur.

After a few attempts I found the sulphate of copper assumed a peculiar tint different from what I had seen while working on the unpurified gas, and, thinking that the sulphuric acid liberated might be in some way acting upon some of the hydrocarbons, I took some of the sulphate out of the tube and treated it with absolute alcohol and also with ether, and obtained a peculiar oily substance, apparently a mixture of hydrocarbons, but have not as yet been able to obtain a sufficient quantity of it to make any experiments upon it, but hope to be able to do so before long. This was sufficient to prove to me what caused the increase in weight of the U-tube and showed that it was not due to sulphuretted hydrogen.

I have not yet had an opportunity to try this method on unpurified coal gas, but according to Mr. Wright's statements it gives very satisfactory results.

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## ON THE FRACTIONAL DE-HYDRATION OF AMMONIUM ALUM, AND THE ATOMICITY OF ALUMINUM.

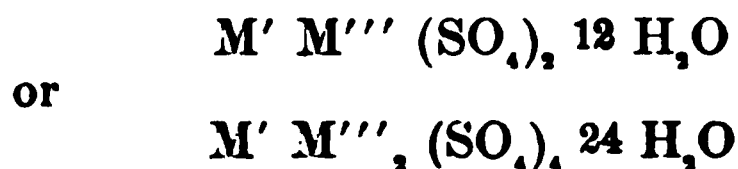
BY J. W. MALLET.

In the course of experiments preparatory to my investigation of the atomic weight of aluminum,\* I examined the behavior of crystallized ammonium-aluminum alum when dried under various conditions, among others when placed over sulphuric acid in a desiccator.

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\* Philosophical Transactions, 1880, Part III.

The question of the extent to which water of crystallization is removable, under well-defined conditions, from the alums, bears upon the adoption of the formula



as correctly representing one of these salts. This question has been discussed by Lupton and Watts,† on the basis of the experiments of Hertwig;‡ Lupton§ and Heintz,|| with the general result that in sundry cases the amounts of water lost, by heating to definite points of temperature, or by evaporation of a solution mixed with excess of sulphuric acid, point to the second of these formulæ, from which odd numbers—such as 19, 21 and 23—of molecules of water may be withdrawn, and which therefore cannot be halved and thus reduced to the simpler formula if but a single molecule of the anhydrous salt be supposed to be present in the residue.

Having ascertained that ammonium-aluminum alum loses largely of its water of crystallization by exposure to air of ordinary atmospheric temperature and pressure dried by a surface of sulphuric acid, and that the loss, even after it has become very slow, goes on for a very long time, I determined, as a separate question from that with which I was chiefly occupied, to continue the drying under these conditions until loss of weight should cease, and see what number of molecules of water would be left. The experiment lasted more than *two years*, weighings being made at first at intervals of a few hours, then of a day, of two days, of two weeks, and, toward the end, of a month. The temperature of the room, which in winter was warmed continuously by night as well as day, ranged generally about from 20° to 27° C., but occasionally passed these limits to a considerable extent during long-continued periods of hotter or colder weather out of doors, and such variations of temperature as occurred made themselves perceptible in the more or less rapid loss of weight of the salt experimented on. The alum was in the form of a fine crystalline meal, spread out to the depth of 7 or 8 millimeters over the surface of a platinum capsule about

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† Watts' Dictionary of Chemistry, 3d Suppl., Part I., pp. 65, 66.

‡ Pogg. Ann., lv., 99.

§ Chem. Soc. Jour. (Lond.) [2], xiii., 201.

| Pogg. Ann., iv., 331.

10 cm. in diameter. The desiccator was of large size, and furnished with a drying tube, containing sulphuric acid, communicating with the outside air, so that the pressure within scarcely differed at any time from that of the atmosphere without.

The following were the results obtained.

Weight of alum at commencement of experiment=39.1654 grams.

Loss after	0 day	9½ hours	—	.0839 gram.
“ “	1 “	2 “	—	.2419 “
“ “	1 “	9 “	—	.3261 “
“ “	2 “	0 “	—	.4423 “
“ “	3 “	2 “	—	.6944 “
				(.7781 “ =1 mol. H <sub>2</sub> O.)
“ “	4 “	4 “	—	.8962 “
“ “	5 “	8½ “	—	1.1703 “
				(1.5562 “ =2 H <sub>2</sub> O.)
“ “	7 “	0 “	—	1.6027 “
“ “	9 “	0 “	—	2.1123 “
				(2.3344 “ =3 H <sub>2</sub> O.)
“ “	11 “	0 “	—	2.5218 “
“ “	13 “	0 “	—	2.9480 “
				(3.1125 “ =4 H <sub>2</sub> O.)
“ “	15 “	4 “	—	3.3821 “
“ “	17 “	0 “	—	3.7365 “
				(3.8906 “ =5 H <sub>2</sub> O.)
“ “	19 “	0 “	—	4.1056 “
“ “	21 “	0 “	—	4.3985 “
				(4.6687 “ =6 H <sub>2</sub> O.)
“ “	23 “	0 “	—	4.6811 “
“ “	25 “	0 “	—	4.8855 “
“ “	27 “	0 “	—	5.1411 “
“ “	29 “	0 “	—	5.3849 “
				(5.4468 “ =7 H <sub>2</sub> O.)
“ “	31 “	0 “	—	5.7015 “
“ “	33 “	0 “	—	6.0237 “
				(6.2250 “ =8 H <sub>2</sub> O.)
“ “	35 “	0 “	—	6.3069 “
“ “	37 “	0 “	—	6.5191 “
“ “	39 “	2 “	—	6.7106 “
				(7.0031 “ =9 H <sub>2</sub> O.)

Loss after 42 day 2 hours—				7.0279 gram.	
				(7.8812	" —10 H <sub>2</sub> O.)
"	"	57	" 23	" — 8.2636	"....
				(8.5593	" —11 H <sub>2</sub> O.)
"	"	73	" 1	" — 9.1001	"
				(9.3374	" —12 H <sub>2</sub> O.)
"	"	103	" 3	" — 9.8668	"
				(10.1156	" —13 H <sub>2</sub> O.)
"	"	133	" 6	" —10.6028	"
				(10.8937	" —14 H <sub>2</sub> O.)
"	"	168	" 7	" —10.9848	"
"	"	198	" 23	" —11.5513	"
				(11.6718	" —15 H <sub>2</sub> O.)
"	"	229	" 23	" —12.2015	"
				(12.4499	" —16 H <sub>2</sub> O.)
"	"	266	" 0	" —12.7269	"
				(13.2380	" —17 H <sub>2</sub> O.)
"	"	298	" 0	" —13.4779	"
				(14.0062	" —18 H <sub>2</sub> O.)
"	"	330	" 22	" —14.0662	"
"	"	364	" 1	" —14.4133	"
"	"	395	" 6	" —14.5237	" (Put fresh acid into
"	"	425	" 23	" —14.5670	distillator.
"	"	467	" 3	" —14.5955	"
"	"	487	" 3	" —14.6146	"
"	"	519	" 3	" —14.6341	"
"	"	549	" 5	" —14.6528	"
"	"	577	" 3	" —14.6675	"
"	"	608	" 6	" —14.6793	"
"	"	639	" 0	" —14.6933	"
"	"	669	" 6	" —14.7277	"
"	"	705	" 1	" —14.7595	"
				(14.7843	" —19 H <sub>2</sub> O.)
"	"	730	" 3	" —14.7847	"
"	"	761	" 3	" —14.7875	"
"	"	800	" 0	" —14.7875	"
"	"	811	" 3	" —14.7858	"
"	"	881	" 23	" —14.7955	"

Hence it appears that by exposure to dried air under the conditions stated, ammonium alum loses 19 out of 24 molecules of

water, and thus adds one case of definite partial de-hydration to those cited by Lupton and Watts\* in favor of the layer formula—the observed residues standing as follows (Watts' table, copied from Lupton's paper, with the case now reported added in brackets):

$(\text{NH}_4)_2\text{Al}_2(\text{SO}_4)_4 \cdot \text{H}_2\text{O}$	$\text{K}_2 \text{ Al}_2 (\text{SO}_4)_4 \cdot \text{H}_2\text{O}$	$(\text{NH}_4)_2\text{Fe}_2(\text{SO}_4)_4 \cdot \text{H}_2\text{O}$	$\text{K}_2 \text{ Fe}_2 (\text{SO}_4)_4 \cdot \text{H}_2\text{O}$
	$\text{K}_2 \text{ Al}_2 (\text{SO}_4)_4 \cdot 3\text{H}_2\text{O}$		$\text{K}_2 \text{ Al}_2 (\text{SO}_4)_4 \cdot 3\text{H}_2\text{O}$
$\left( (\text{NH}_4)_2 \text{ Al}_2 \right) (\text{SO}_4)_4 \cdot 5\text{H}_2\text{O}$	$\text{K}_2 \text{ Al}_2 (\text{SO}_4)_4 \cdot 5\text{H}_2\text{O}$		

If the rate of loss in the interval between the weighings next above and below that corresponding to each molecule be assumed as uniform, it appears that the

1st molecule of water was lost in 85 hours					
2d	“	“	“	78½	“
3d	“	“	“	78½	“
4th	“	“	“	90	“
5th	“	“	“	96	“
6th	“	“	“	122	“
7th	“	“	“	155	“
8th	“	“	“	120	“
9th	“	“	“	182	“
10th	“	“	“	231	“
11th	“	“	“	282	“
12th	“	“	“	459	“
13th	“	“	“	744	“
14th	“	“	“	1121	“
15th	“	“	“	1065	“
16th	“	“	“	1024	“
17th	“	“	“	969	“
18th	“	“	“	964	“
19th	“	“	“	9646	“

Taking into account the changes of atmospheric temperature, especially the more persistent changes due to season, during the time occupied by the experiment, which began on the 3d of August, there seems to have been a gradual decrease in the rate of loss throughout, unmarked by any abrupt variations, except as regards the last molecule of water given off, the removal of which took

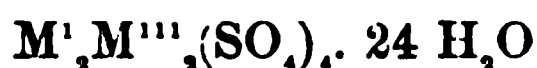
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\* Loc. cit.



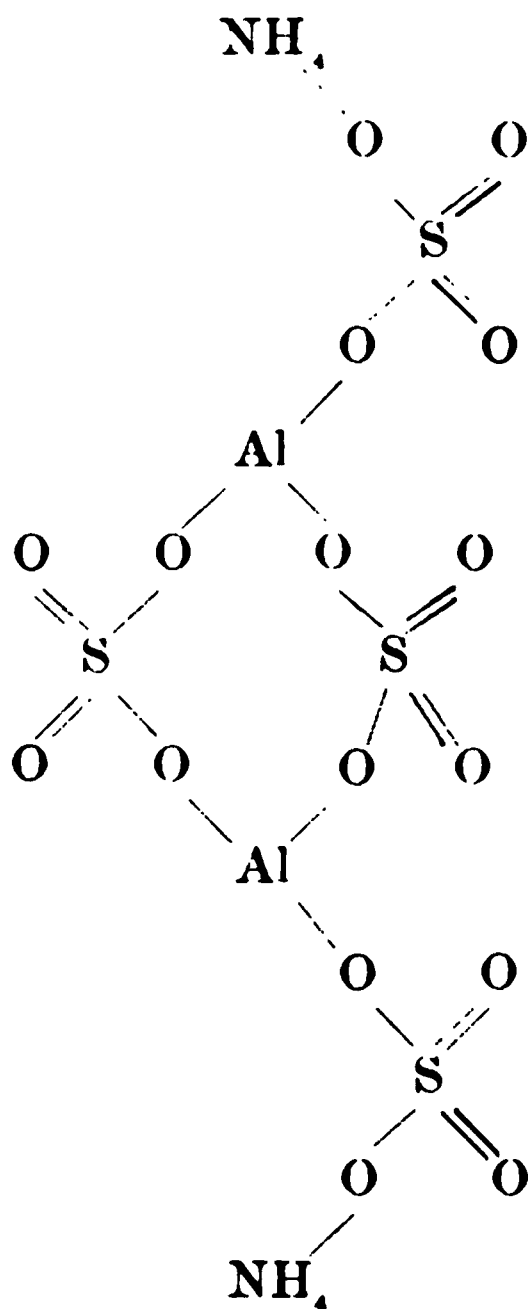
more than a year, and more than as long as all of the 18 preceding molecules.

While this experiment sustains the conclusion of Watts that the proper formula for the alums is:



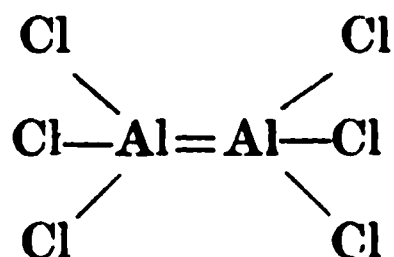
I do not perceive that the further conclusion at which he arrives is valid—viz. “*This being admitted, it follows that aluminum, chromium and iron must be tetrad atomic.*” There is no direct evidence of aluminum exhibiting tetrad character.

All its general relations are those of a triad element. In the alums it is not necessary to assume any “linking function” on the part of the two atoms of aluminum. The constitution of ammonium-aluminum alum, for instance, may be :



If, however, it be assumed that the two aluminum atoms are directly combined, either in this salt or in the haloid compounds of the metal it seems more probable that in such cases there is double linking, and that aluminum is to be considered as triad or *sometimes pentad*. Thus the chloride, taking the for-

mula  $\text{Al}_2\text{Cl}_6$ , as established by Deville and Troost's determination of the vapor density, may be represented as :



This view seems to reconcile the otherwise conflicting evidence as to atomicity derived from the observed vapor densities of aluminum chloride on the one hand and aluminum methid and ethid on the other. The determinations of Buckton and Odling for the vapor densities of the latter of these compounds are quite inconsistent with the common assumption that aluminum is tetrad.

UNIV. OF VIRGINIA, *Sept.* 20, 1882.

## ON NITROSACCHAROSE : A CORRECTION.

BY ARTHUR H. ELLIOTT, PH. B., F. C. S.

In Vol. IV, p. 147 of this Journal, will be found a paper by me on nitrosaccharose, and at the end of said paper some theoretical conclusions are given which I desire to withdraw.

When the paper was written I was about starting on my summer vacation, and I did not see it again until after it was printed, and a second reading of it showed me I had made a mistake in my figures.

Prof. C. F. Chandler also kindly called my attention to the improbability of the formula given by me to nitrosaccharose. But in assigning the formula given, I was led into error by a corresponding formula found in the English edition of Wislicenus Strecker's handbook, where the octacetate of saccharose is given as  $\text{C}_{12}\text{H}_{14}(\text{C}_2\text{H}_3\text{O}_2)_8\text{O}_{11}$ , the correct formula being  $\text{C}_{12}\text{H}_{14}(\text{O.C}_2\text{H}_3\text{O})_8\text{O}_8$ , eight molecules of the group  $\text{C}_2\text{H}_3\text{O}$  replacing the hydrogen in eight molecules of hydroxyl ; thus giving saccharose the formula  $\text{C}_6\text{H}_7(\text{OH})_4\text{O}-\text{O}-\text{O}(\text{OH})_4\text{C}_6\text{H}_7$ .

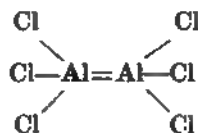
In regard to the error in my figures, I took the percentage of of cane sugar found and added it to the percentage of  $\text{NO}_2$ . Correctly, I should have taken the percentage of cane sugar minus the hydroxyl, and added this residue  $(\text{C}_{12}\text{H}_{14}\text{O}_8)$  to the  $\text{NO}_2$  found. By

doing this 28.55, the percentage of cane sugar found is equal to 17.19 per cent. of  $C_{12}H_{22}O_{11}$ ; which added to the  $NO_2$  found (41.45) gives a total percentage of 58.64; and the ratio of cane sugar to  $NO_2$  becomes 1 to 8.028 instead of 1 to 8.009, as given before. Practically the ratio of the molecules of cane sugar and  $NO_2$  remain the same, 1 to 8.

From a consideration of this relation and of the formula for saccharose quoted above, the true formula for nitrosaccharose becomes  $C_{12}H_{22}(O.NO_2)_8O$ , or an octonitrate; thus coming into the same class of organic nitrates as nitrocellulose and nitroglycerine.

SCHOOL OF MINES, *Sept.* 1882.

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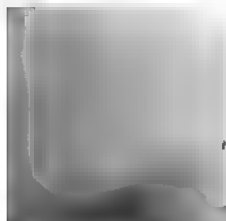
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In regard to the error in my figures, I took the percentage of cane sugar found and added it to the percentage of  $\text{NO}_2$ . Correctly, I should have taken the percentage of cane sugar minus the hydroxyl, and added this residue ( $\text{C}_{12}\text{H}_{11}\text{O}_5$ ) in the  $\text{NO}_2$  found. By

doing this 28.55, the percentage of cane sugar found is equal to 17.19 per cent. of  $C_{12}H_{22}O_{11}$ ; which added to the  $NO_2$  found (41.45) gives a total percentage of 58.64; and the ratio of cane sugar to  $NO_2$  becomes 1 to 8.028 instead of 1 to 8.009, as given before. Practically the ratio of the molecules of cane sugar and  $NO_2$  remain the same, 1 to 8.

From a consideration of this relation and of the formula for saccharose quoted above, the true formula for nitrosaccharose becomes  $C_{12}H_{22}(O.NO_2)_8O_8$ , or an octonitrate; thus coming into the same class of organic nitrates as nitrocellulose and nitroglycerine.

SCHOOL OF MINES, *Sept.* 1882.



or very nearly so, but when the sulphur has not been completely oxydized the liquid will have a decidedly yellow color.

With the finished gas the solution was always colorless or very nearly so, but when I attempted to estimate the sulphur in the unpurified gas, either before or after enriching, I always obtained a deeply-colored liquid, and the total amount of sulphur but little more than the amount obtained from the purified gas, and much less than I had reason to suppose existed. Why this should be, I am as yet unable to explain, but as it was actually the case, after a few repeated attempts I gave it up and started to make estimations of the amount of sulphuretted hydrogen, thinking if I could accomplish this it might give me some idea of the sulphur.

I tried making some determinations in an absorption tube for gas analysis, using binocide of manganese as recommended by Prof. Bunsen, but I soon found that in cases like this where the quantity of sulphur was comparatively small and the amount of gas which could be used was also very small, the liability to error was so great that no satisfactory results could be obtained. I then concluded to try it by absorption in a U-tube in order to employ a large volume of gas. At first liquids were used, such as solution of acetate of lead, sulphate of copper, etc., but none of these worked well. At the suggestion of Mr. Lees, I tried dehydrated sulphate of copper, this absorbed the sulphuretted hydrogen very nicely, forming sulphide of copper and sulphuric acid; but the difficulty was that in order to completely dehydrate it, it was necessary to pulverize it, and in so doing, it generally became so finely powdered that it would pack in the U-tube and clog it up until the pressure would become sufficient to blow up through it, forming direct gas passages, through which the gas would pass and hardly be acted upon. About this time I was busily engaged with some other work and had no more time to devote to the sulphur determinations and left them for awhile. In the meantime, which was about the first of April, Mr. Lewis T. Wright, of England, published an article in the *English Gas Light Journal* on the estimation of sulphuretted hydrogen and carbonic acid in coal gas. In this article he mentioned the difficulty offered to the passage of the gas in the U-tube by the ordinary dehydrated sulphate of copper, and said he had overcome this by drying it first at about  $100^{\circ}\text{C}$ ., then adding a little water to it and stirring it with a glass rod in order to slake it, which makes it granular, then drying it again at  $100^{\circ}\text{C}$ . and then at  $155^{\circ}\text{C}$ . At my first opportunity I prepared some in this manner and found it porous, and

it worked very nicely, completely overcoming the previous difficulty.

In order to give a better idea of the method employed, I will describe the apparatus which was used. It consisted first of a drying cylinder containing fused chloride of calcium, next to this was attached a weighed six-inch glass stoppered U tube containing the dehydrated sulphate of copper, a bit of cotton was put on the top of the sulphate in the U-tube to prevent any of it being blown out. To this U-tube was attached another weighed U-tube containing soda-lime and chloride of calcium to catch any carbonic acid and thereby enable me to determine it at the same time, and after this was placed, a meter to measure the quantity of gas passed through the tubes, and from the meter was a tube leading to a small gas burner where the gas was burnt.

The object of the first chloride of calcium was to dry the gas, for any moisture would be caught in the sulphate of copper tube and weighed as sulphuretted hydrogen. In burning the gas in the Referee's apparatus, I had to pass it through a wet meter first, to measure it, and in order to avoid a loss by the sulphur being washed out by the water, the water was replaced by glycerine and gas allowed to pass through it for a long time before using it, but in this case I was able to place the meter after my absorption apparatus, and thus avoid any error from this cause.

The apparatus was first tried on the pipe leading from the hydrogen main; after letting a sufficient quantity blow out to ensure that it was fresh gas, for when the gas was first turned on it failed to produce any stain on acetate of lead paper showing that the sulphur was being removed from the gas by the iron rust in the pipe. Sometime after it began to produce a stain, the apparatus was attached by means of a T-shaped pipe so that the gas could be kept burning all the time and thus have the flow of gas up the iron pipe much more rapid, and thereby have it as little acted upon as possible.

The gas was allowed to pass very slowly through the apparatus, not over one foot in three hours; after one foot had passed, the U-tube was detached and weighed, and found to have increased 0.198 grammes in weight, which would give 315.51 grains of sulphuretted hydrogen in 100 cubic feet, or 0.47 per cent. by volume. After this, four more determinations were made, allowing only one-half foot to pass each time, and although in this case the results had to be multiplied by 200 they agreed very closely, as may be seen by the following table. The column marked A gives the increase

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in grammes of weight of the U-tube, B gives the number of grains of sulphuretted hydrogen in 100 cubic feet, and C the percentage :

	A.	B.	C.
No. 1.....	.1085	344.91	0.51 per cent.
No. 2.....	.1100	339.52	0.52 “ “
No. 3.....	.1110	342.59	0.52 “ “
No. 4.....	.1105	341.00	0.52 “ “

These I consider as agreeing very closely.

A few days after I began the experiments upon the finished gas and was surprised to find that the U-tube gained more in weight than it did when using the unpurified, sometimes showing as much as 100 grains more in 100 cubic feet, while the Referee's apparatus, as used by Dr. Love and also by myself, did not give over 10 grains of total sulphur.

After a few attempts I found the sulphate of copper assumed a peculiar tint different from what I had seen while working on the unpurified gas, and, thinking that the sulphuric acid liberated might be in some way acting upon some of the hydrocarbons, I took some of the sulphate out of the tube and treated it with absolute alcohol and also with ether, and obtained a peculiar oily substance, apparently a mixture of hydrocarbons, but have not as yet been able to obtain a sufficient quantity of it to make any experiments upon it, but hope to be able to do so before long. This was sufficient to prove to me what caused the increase in weight of the U-tube and showed that it was not due to sulphuretted hydrogen.

I have not yet had an opportunity to try this method on unpurified coal gas, but according to Mr. Wright's statements it gives very satisfactory results.

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## ON THE FRACTIONAL DE-HYDRATION OF AMMONIUM ALUM, AND THE ATOMICITY OF ALUMINUM.

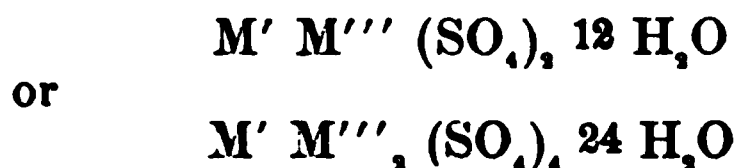
BY J. W. MALLET.

In the course of experiments preparatory to my investigation of the atomic weight of aluminum,\* I examined the behavior of crystallized ammonium-aluminum alum when dried under various conditions, among others when placed over sulphuric acid in a desiccator.

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\* Philosophical Transactions, 1880, Part III.

The question of the extent to which water of crystallization is removable, under well-defined conditions, from the alums, bears upon the adoption of the formula



as correctly representing one of these salts. This question has been discussed by Lupton and Watts,† on the basis of the experiments of Hertwig;‡ Lupton§ and Heintz,|| with the general result that in sundry cases the amounts of water lost, by heating to definite points of temperature, or by evaporation of a solution mixed with excess of sulphuric acid, point to the second of these formulæ, from which odd numbers—such as 19, 21 and 23—of molecules of water may be withdrawn, and which therefore cannot be halved and thus reduced to the simpler formula if but a single molecule of the anhydrous salt be supposed to be present in the residue.

Having ascertained that ammonium-aluminum alum loses largely of its water of crystallization by exposure to air of ordinary atmospheric temperature and pressure dried by a surface of sulphuric acid, and that the loss, even after it has become very slow, goes on for a very long time, I determined, as a separate question from that with which I was chiefly occupied, to continue the drying under these conditions until loss of weight should cease, and see what number of molecules of water would be left. The experiment lasted more than *two years*, weighings being made at first at intervals of a few hours, then of a day, of two days, of two weeks, and, toward the end, of a month. The temperature of the room, which in winter was warmed continuously by night as well as day, ranged generally about from 20° to 27° C., but occasionally passed these limits to a considerable extent during long-continued periods of hotter or colder weather out of doors, and such variations of temperature as occurred made themselves perceptible in the more or less rapid loss of weight of the salt experimented on. The alum was in the form of a fine crystalline meal, spread out to the depth of 7 or 8 millimeters over the surface of a platinum capsule about

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† Watts' Dictionary of Chemistry, 3d Suppl., Part I., pp. 65, 66.

‡ Pogg. Ann., iv., 99.

§ Chem. Soc. Jour. (Lond.) [2], xiii., 201.

| Pogg. Ann., iv., 331.

10 cm. in diameter. The desiccator was of large size, and furnished with a drying tube, containing sulphuric acid, communicating with the outside air, so that the pressure within scarcely differed at any time from that of the atmosphere without.

The following were the results obtained.

Weight of alum at commencement of experiment=39.1654 grams.

Loss after	0	day	9½	hours	—	.0839	gram.
" "	1	"	2	"	—	.2419	"
" "	1	"	9	"	—	.3261	"
" "	2	"	0	"	—	.4423	"
" "	3	"	2	"	—	.6944	"
						(.7781	" =1 mol. H <sub>2</sub> O.)
" "	4	"	4	"	—	.8962	"
" "	5	"	8½	"	—	1.1703	"
						(1.5562	" =2 H <sub>2</sub> O.)
" "	7	"	0	"	—	1.6027	"
" "	9	"	0	"	—	2.1123	"
						(2.3344	" =3 H <sub>2</sub> O.)
" "	11	"	0	"	—	2.5218	"
" "	13	"	0	"	—	2.9480	"
						(3.1125	" =4 H <sub>2</sub> O.)
" "	15	"	4	"	—	3.3821	"
" "	17	"	0	"	—	3.7365	"
						(3.8906	" =5 H <sub>2</sub> O.)
" "	19	"	0	"	—	4.1056	"
" "	21	"	0	"	—	4.3985	"
						(4.6687	" =6 H <sub>2</sub> O.)
" "	23	"	0	"	—	4.6811	"
" "	25	"	0	"	—	4.8855	"
" "	27	"	0	"	—	5.1411	"
" "	29	"	0	"	—	5.3849	"
						(5.4468	" =7 H <sub>2</sub> O.)
" "	31	"	0	"	—	5.7015	"
" "	33	"	0	"	—	6.0237	"
						(6.2250	" =8 H <sub>2</sub> O.)
" "	35	"	0	"	—	6.3069	"
" "	37	"	0	"	—	6.5191	"
" "	39	"	2	"	—	6.7106	"
						(7.0031	" =9 H <sub>2</sub> O.)

Loss after 42 day 2 hours—— 7.0279 gram.

					(7.8812	" =10 H <sub>2</sub> O.)
"	"	57	"	23	" ——— 8.2636	"....
					(8.5593	" =11 H <sub>2</sub> O.)
"	"	73	"	1	" ——— 9.1001	"
					(9.3374	" =12 H <sub>2</sub> O.)
"	"	103	"	3	" ——— 9.8668	"
					(10.1156	" =13 H <sub>2</sub> O.)
"	"	133	"	6	" ———10.6028	"
					(10.8937	" =14 H <sub>2</sub> O.)
"	"	168	"	7	" ———10.9848	"
"	"	198	"	23	" ———11.5513	"
					(11.6718	" =15 H <sub>2</sub> O.)
"	"	229	"	23	" ———12.2015	"
					(12.4499	" =16 H <sub>2</sub> O.)
"	"	266	"	0	" ———12.7269	"
					(13.2380	" =17 H <sub>2</sub> O.)
"	"	298	"	0	" ———13.4779	"
					(14.0062	" =18 H <sub>2</sub> O.)
"	"	330	"	22	" ———14.0662	"
"	"	364	"	1	" ———14.4133	"
"	"	395	"	6	" ———14.5237	" [Put fresh acid into desiccator.
"	"	425	"	23	" ———14.5670	"
"	"	467	"	3	" ———14.5955	"
"	"	487	"	3	" ———14.6146	"
"	"	519	"	3	" ———14.6341	"
"	"	549	"	5	" ———14.6528	"
"	"	577	"	3	" ———14.6675	"
"	"	608	"	6	" ———14.6793	"
"	"	639	"	0	" ———14.6933	"
"	"	669	"	6	" ———14.7277	"
"	"	705	"	1	" ———14.7595	"
					(14.7843	" =19 H <sub>2</sub> O.)
"	"	730	"	2	" ———14.7847	"
"	"	761	"	3	" ———14.7875	"
"	"	800	"	0	" ———14.7875	"
"	"	811	"	3	" ———14.7858	"
"	"	998	"	23	" ———14.7955	"

Hence it appears that by exposure to dried air under the conditions stated, ammonium alum loses 19 out of 24 molecules of

water, and thus adds one case of definite partial de-hydration to those cited by Lupton and Watts\* in favor of the layer formula—the observed residues standing as follows (Watts' table, copied from Lupton's paper, with the case now reported added in brackets):

$(\text{NH}_4)_2\text{Al}_2(\text{SO}_4)_4 \cdot \text{H}_2\text{O}$	$\text{K}_2 \text{ Al}_2 (\text{SO}_4)_4 \cdot \text{H}_2\text{O}$	$(\text{NH}_4)_2\text{Fe}_2(\text{SO}_4)_4 \cdot \text{H}_2\text{O}$	$\text{K}_2 \text{ Fe}_2 (\text{SO}_4)_4 \cdot \text{H}_2\text{O}$
	$\text{K}_2 \text{ Al}_2 (\text{SO}_4)_4 \cdot 3\text{H}_2\text{O}$		$\text{K}_2 \text{ Al}_2 (\text{SO}_4)_4 \cdot 3\text{H}_2\text{O}$
$\left( (\text{NH}_4)_2 \text{ Al}_2 \right. \\ \left. (\text{SO}_4)_4 \cdot 5\text{H}_2\text{O} \right)$	$\text{K}_2 \text{ Al}_2 (\text{SO}_4)_4 \cdot 5\text{H}_2\text{O}$		

If the rate of loss in the interval between the weighings next above and below that corresponding to each molecule be assumed as uniform, it appears that the

1st molecule of water was lost in 85 hours					
2d	•	“	“	“	78½ “
3d		“	“	“	78½ “
4th		“	“	“	90 “
5th		“	“	“	96 “
6th		“	“	“	122 “
7th		“	“	“	155 “
8th		“	“	“	120 “
9th		“	“	“	182 “
10th		“	“	“	231 “
11th		“	“	“	282 “
12th		“	“	“	459 “
13th		“	“	“	744 “
14th		“	“	“	1121 “
15th		“	“	“	1065 “
16th		“	“	“	1024 “
17th		“	“	“	969 “
18th		“	“	“	964 “
19th		“	“	“	9646 “

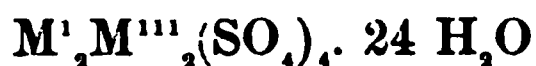
Taking into account the changes of atmospheric temperature, especially the more persistent changes due to season, during the time occupied by the experiment, which began on the 3d of August, there seems to have been a gradual decrease in the rate of loss throughout, unmarked by any abrupt variations, except as regards the last molecule of water given off, the removal of which took

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\* Loc. cit.

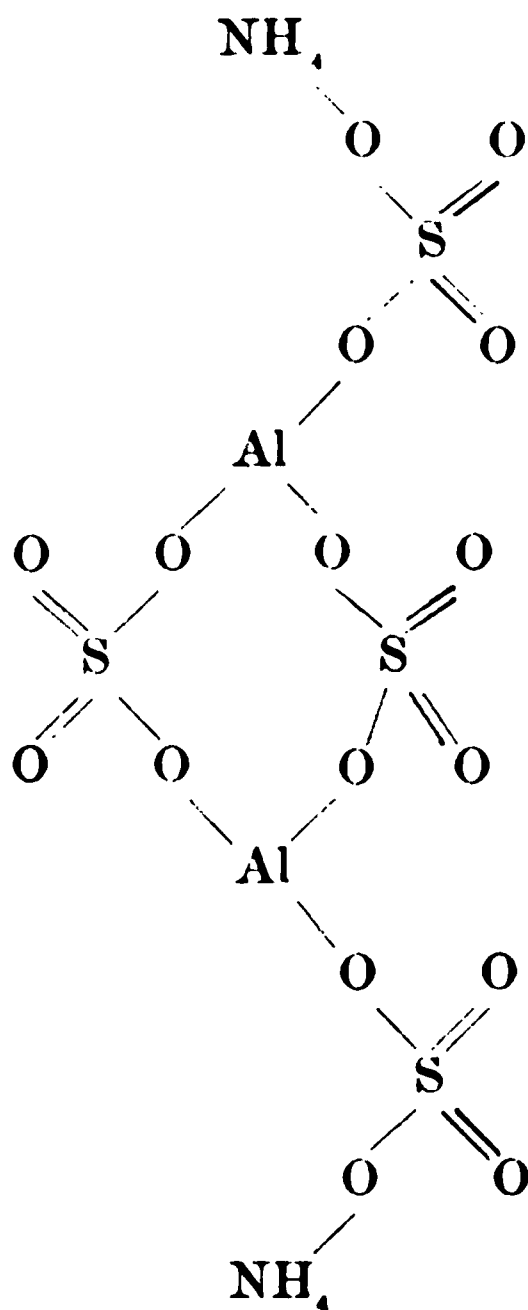
more than a year, and more than as long as all of the 18 preceding molecules.

While this experiment sustains the conclusion of Watts that the proper formula for the alums is:



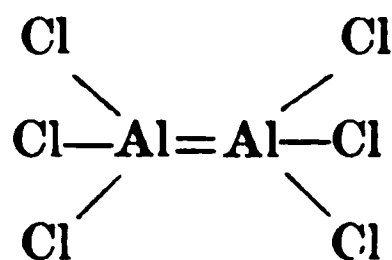
I do not perceive that the further conclusion at which he arrives is valid—viz. “*This being admitted, it follows that aluminum, chromium and iron must be tetrad atomic.*” There is no direct evidence of aluminum exhibiting tetrad character.

All its general relations are those of a triad element. In the alums it is not necessary to assume any “linking function” on the part of the two atoms of aluminum. The constitution of ammonium-aluminum alum, for instance, may be :



If, however, it be assumed that the two aluminum atoms are directly combined, either in this salt or in the haloid compounds of the metal it seems more probable that in such cases there is double linking, and that aluminum is to be considered as triad or *sometimes pentad*. Thus the chloride, taking the for-

mula  $\text{Al}_2\text{Cl}_6$  as established by Deville and Troost's determination of the vapor density, may be represented as :



This view seems to reconcile the otherwise conflicting evidence as to atomicity derived from the observed vapor densities of aluminum chloride on the one hand and aluminum methid and ethid on the other. The determinations of Buckton and Odling for the vapor densities of the latter of these compounds are quite inconsistent with the common assumption that aluminum is tetrad.

UNIV. OF VIRGINIA, *Sept.* 20, 1882.

## ON NITROSACCHAROSE : A CORRECTION.

BY ARTHUR H. ELLIOTT, PH. B., F. C. S.

In Vol. IV, p. 147 of this Journal, will be found a paper by me on nitrosaccharose, and at the end of said paper some theoretical conclusions are given which I desire to withdraw.

When the paper was written I was about starting on my summer vacation, and I did not see it again until after it was printed, and a second reading of it showed me I had made a mistake in my figures.

Prof. C. F. Chandler also kindly called my attention to the improbability of the formula given by me to nitrosaccharose. But in assigning the formula given, I was led into error by a corresponding formula found in the English edition of Wislicenus Strecker's handbook, where the octacetate of saccharose is given as  $\text{C}_{12}\text{H}_{14}(\text{C}_2\text{H}_3\text{O}_2)_8\text{O}_{11}$  the correct formula being  $\text{C}_{12}\text{H}_{14}(\text{O}.\text{C}_2\text{H}_3\text{O})_8\text{O}_8$  eight molecules of the group  $\text{C}_2\text{H}_3\text{O}$  replacing the hydrogen in eight molecules of hydroxyl ; thus giving saccharose the formula  $\text{C}_6\text{H}_7(\text{OH})_4\text{O}-\text{O}-\text{O}(\text{OH})_4\text{C}_6\text{H}_7$ .

In regard to the error in my figures, I took the percentage of cane sugar found and added it to the percentage of  $\text{NO}_2$ . Correctly, I should have taken the percentage of cane sugar minus the hydroxyl, and added this residue  $(\text{C}_{12}\text{H}_{14}\text{O}_8)$  to the  $\text{NO}_2$  found. By



doing this 28.55, the percentage of cane sugar found is equal to 17.19 per cent. of  $C_{12}H_{22}O_{11}$ ; which added to the  $NO_2$  found (41.45) gives a total percentage of 58.64; and the ratio of cane sugar to  $NO_2$  becomes 1 to 8.028 instead of 1 to 8.009, as given before. Practically the ratio of the molecules of cane sugar and  $NO_2$  remain the same, 1 to 8.

From a consideration of this relation and of the formula for saccharose quoted above, the true formula for nitrosaccharose becomes  $C_{12}H_{22}(O.NO_2)_8O$ , or an octonitrate; thus coming into the same class of organic nitrates as nitrocellulose and nitroglycerine.

SCHOOL OF MINES, *Sept.* 1882.

## REVIEW OF PAPERS ON ANALYTICAL CHEMISTRY (PROXIMATE) WHICH HAVE APPEARED DURING THE YEAR 1882.

BY E. WALLER, PH. D.

**MILK ANALYSIS.**—To determine WATER, *Marpmann* (*Biedermann's Centrbl. fur Agrikult. Chem* XI., pt. 2) proposes to fill a calcium chloride tube with cotton wool, then to saturate the cotton with the milk, and to dry by drawing a stream of hot dry air through the tube. The FAT may also be determined in the same sample by extraction with benzol or ether.

*O. Hehner* gives the results of experiments on the methods for milk analysis adopted by the Soc. of Pub. Anal. (*Analyst*, VII., 60). In determining WATER the operation was more rapid on the water bath than in an oven. After drying for three hours an amount varying from 0.06 to 0.29 % may be driven out by further heating. A temperature above 100° C. apparently causes decomposition and loss of solids not fat. For BUTTER FAT the Soxhlet apparatus removes about 0.2 % more than boiling out with three or more successive quantities of ether. Nothing is gained by granulating the milk residue, by allowing the ether to act for a long time, or by adding 10 % absolute alcohol to it. Portions of the ASH are volatile at red heat.

*A. Mayer* states that the oil from ARTIFICIAL BUTTER has a much lower specific gravity than that from natural butter. (*Fuehling's Landw. Ztg.* XXXI., 92)

The amount of COPPER NORMALLY PRESENT IN WHEAT is stated by *J. van D. Berghe* as 0.008 to 0.01 % (*Les Mondes*, I., [3] 209).

TO TEST BREAD FOR THE PRESENCE OF ALUM, *A. W. Blyth* (*Analyst*, VII., 16) proposes the use of slips of gelatine. The bread is crumbled into a vessel, a couple of slips of dry commercial gelatine placed with it, and distilled water added sufficient to cover the materials. On standing over night, the alum concentrates in the gelatine, and when dipped in fresh tincture of logwood, to which an equal bulk of 10 % solution of ammonium carbonate has been added the slips will dye blue if alum was present. When alum is not present, the slip becomes reddish brown, the tint being removable by soaking in glycerin. Experiments upon alumed bread seemed to show that much of the alum added remains as such after baking. Nearly all, if not quite all, of the alumina added as alum may be removed from the bread by soaking it in a 5 % HCl. solution.

IN EXAMINING TEA, *J. Carter Bell* (*Analyst*, VII., 7) gives the results of the examination of 58 samples. The variations were as follows :

Per cent. of ash.....	5.64 to 8.58
Per cent. of ash soluble in water.....	32.70 to 67.41
Per cent. alkalinity of ash ( $=K_2O$ ).....	14.24 to 30.57

IN EXAMINING ROASTED COFFEE, *A. Smetham* (*Analyst*, VII., 73) concludes from the examination of seven genuine samples, first, that moisture (loss at  $100^{\circ}C$ .) should always be first determined. The per cent. of "crude fibre" is the best guide where chicory is the adulterant. The determination of oil, cellulose, ash (soluble and insoluble), nitrogen, and sp. gr. of the 10 % infusion, are principally of service as checks on the work.

The crude fibre was found to vary between 73.71 and 75.7 %, referred to the dry samples. It was determined by boiling 2 gms. of the samples with three successive lots of water (100 cc. each), and finally washing on a tared filter until the washings were colorless.

FOR COLORIMETRIC ESTIMATION OF SALICYLIC ACID in articles of food, urine, &c., *MM. Pellet and De Grobert* (*Revue des Industries*, No. 53, 1882) propose ferric chloride solution (sp. gr. 1.005 to 1.008) containing 0.5 gms. free acid per 100 cc. In testing butter by this method, 25 gms. are shaken with 5 or 6 drops sulphuric acid (of  $30^{\circ}B$ .) and 75 cc. benzol. The total volume is then made up to some convenient bulk (100 cc.) It is then filtered, and 5 or 10 cc. mixed with the same bulk of water, and a few drops of the ferric chloride solution added. The mixture is gently shaken, the production of an emulsion being avoided, and the tint compared with that obtained in a standard solution.

EXAMINATION OF WINES. General methods are given by *J. Nessler and M. Barth* (*Fres. Zeitz. Anal. Chem.* XXI., 43) and *L. Roesler* (*Chem. Zeit.*, VI., 306).

FOR EXTRACT, the first named authors find that evaporation at  $100^{\circ}C$ . for three hours does not remove the glycerin. Some loss by decomposition of non-volatile acids will be experienced, unless a known bulk of  $Ba(HO)_2$  solution is added. For genuine wines, the extract less acids is rarely under 10 %. *Roesler* asserts that evaporation at temperatures above  $80^{\circ}$  causes both loss of glycerin and decomposition of the residue. He recommends evaporation at  $60^{\circ}C$ . in partial vacuum.

For added SUGAR *Nessler and Barth* prefer a modification of Neubauer's polariscope test. Cane sugar if present is partially or wholly inverted by the acids of the wine on evaporating. Caramel has no optical effect. *Roesler* decolorizes with bone black and uses the Fehling test.

For FREE TARTARIC ACID *Nessler and Barth* evaporate a portion, add alcohol until no further precipitation ensues, filter, evaporate off the alcohol, add water, clarify by bone black, and then add one or two cc. of 20% solution of calcium acetate. The amount of free tartaric acid should not be over one-sixth the amount of the non-volatile acids.

*Roesler* first determines tartar by adding 50 cc. ether to 10 cc. of wine, allowing to stand 24 hours, washing with ether, dissolving in boiling water and titrating. Then 10 cc. of the wine is neutralized with standard KOH solution, 40 cc. of the wine added, and 10 cc. of the mixture treated as above. The excess of the acidity in the second case is due to free tartaric acid.

*Nessler and Barth* also state that the ASH should be at most 1.4%. In some cases the wines may have been fraudulently diluted and the percentage of ash increased by use of SALT. In such a case the ash does not readily burn white. To detect this fraud, the wine is decolorized by bone black and Volhard's method used. The usual amount of Cl in wines is under 0.002%, the maximum 0.005%.

The last named authors also state that the maximum amount of CITRIC ACID in genuine wines is 0.002 to 0.003 per cent., but that acid is frequently added as such or in tamarinds (which contain 13.5%.

To determine it, 100 cc. of the wine are evaporated to 7 cc., cooled, the soluble portion extracted by alcohol (of 80%) the residue diluted to 20 cc., *partly* neutralized with milk of lime, (for red wines bone black is also added at this point) filtered, diluted to 100 cc., and the citric and tartaric acids separated by about one cc. of neutral lead acetate. From this combination the lead is separated by agitation with  $H_2S$  water, and filtering. After removing the  $H_2S$ , lime is added to render the solution alkaline, and after filtering it is made slightly acid with acetic acid and allowed to stand to separate the calcium tartrate. In the filtrate the acetic acid is expelled by heat and the solution is finally boiled until the calcium citrate separates in crystalline form when it is filtered and weighed. *Roesler* determines the GRAVITY preferably by Sprengel's pyknometer; ALCOHOL by distilling off two-thirds, making the distillate up to the

original volume and taking the gravity; TANNIC ACID by distilling off the alcohol, making up to the original volume, agitating with bone black, filtering, and applying Neubauer's modification of Löwenthal's method, (permanganate and indigo carmine); ACETIC ACID by adding baryta hydrate, distilling off alcohol, and then distilling with phosphoric acid (Kessel-Neubauer's method); GLYCERIN by evaporating (100 cc.) down to one-third, rendering slightly alkaline with slaked lime, evaporating to dryness, extracting with alcohol, evaporating this again to dryness and extracting the glycerin from the residue by a mixture of alcohol and ether, (Neubauer-Reichardt's method); and NITROGENOUS CONSTITUENTS by evaporating 10 to 20 cc. in a thin glass vessel, pulverizing residue glass and all, and making a combustion as usual.

For TANNIN AND CÆNOGALLIC ACID, *F. Jean* (*Comptes Rend.* XCIV. No. 11) evaporates 50 to 100 cc. down to small bulk—adds dry precipitated silica, dries at 60 to 70°C., extracts with ether containing a little hydrochloric acid, evaporates the ethereal solution, and dissolves in water, up to the original bulk. In 10 cc. of this solution the combined acids are determined by titrating with iodine, and in the remainder the cœnogallic acid is determined (after shaking with rasped hide to remove tannin) by titration again with iodine.

For GLYCERINE in sweet wines *E. Borgmann* (*Fres. Zeits. Anal. Chem.*, XXI, 239) evaporates 100 cc. to a syrup, extracts the residue with 100 cc. of absolute alcohol, and then adds 150 cc. of ether. The sugar settles to the bottom, leaving the glycerine in solution.

For RED COLORING MATTERS *J. De Groot* (*Repert der Analyt. Chem.*, II, 95) adds to a wine or other liquid to be tested, one volume chloroform and three volumes absolute alcohol, and agitates briskly; three volumes of distilled water are then carefully added without shaking. Many coloring matters separate at the point where the two liquids are in contact. A few, as, for instance, fuchsin, pass into the chloroform. By the manner of separation or the coloration imparted to one or both liquids, many of the coloring matters may be identified. To detect BORDEAUX RED IN WINES *C. Thomas* (*Chem. Zeit.*, VI, 244) precipitates the wine with barium hydrate—filters—removes the excess of barium by ammonium carbonate, and in the filtrate by evaporation, ignition, etc.; tests for the presence of sulphuric acid arising from the presence of a sulpho compound. *M. Guichard* (*Journ. de Pharm. et de Chim.*, May 1882) tests for the same coloring matter by dyeing silk with the wine. The natural coloring matter gives only a violet or lilac

shade, which turns greenish with ammonia, while the Bordeaux red dyes the silk a garnet shade, turning darker with ammonia; also a drop of genuine wine let fall on a lump of chalk moistened with alum solution gives a violet gray spot; whereas, if Bordeaux red is present, a red stain appears.

*F. Musculus* and *C. Anthor* (*Repert. Analyt. Chem.*, II, 142) find that the natural WINES OF A BAD YEAR contain but little alcohol and much acid, but contain more ash, extractive and phosphoric acid than even the best wines of a good year.

For SULPHUROUS ACID IN WINES *B. Haas* (*Ber. D. Chem. Ges.*, XV, 154) distils off about half of a measured quantity in a current of  $\text{CO}_2$  into a bulb tube containing iodine solution, when the sulphuric acid formed may be determined by  $\text{BaCl}_2$ . *L. Lieberman* (*Ber. D. Chem. Ges.*, XV, 439) distils in a similar manner, oxidizing by the use of  $\text{HNO}_3$ . The last-named author makes a qualitative test upon the distillate by using iodic acid and chloroform. A small amount of  $\text{SO}_2$  gives the violet coloration to the chloroform; larger amounts a yellowish brown.

OILS, &c.—To distinguish between OLIVE AND COTTON SEED OIL *M. Zecchim* (*Les Mondes*, May 13, 1882) agitates 5 cc. of the oil with 10 cc. of colorless nitric acid (sp. gr. 1.4), and then allows it to stand for five or six minutes. Pure olive oil will then appear a light ash gray color, with a slight yellow reflex; after some time only it becomes dark colored. Cottonseed oil becomes golden yellow, soon turning coffee brown. Mixtures of the two give intermediate shades.

To detect ROSIN OR PARAFFIN OIL IN LARD, TALLOW, ETC., *W. Rodiger* (*Chem. Zeit.*, 1882, 118) saponifies and then extracts with benzine. If much soda lye is necessary to cause the separation of the soap from its solution in water, palm or cocoanut oil is probably present.

To determine ROSIN IN FATS, *T. S. Gladding* (*Am. Chem. Jour.*, III, 416), dissolves about 0.5 gm. in 20 cc. alcohol (95 %) and saponifies by alcoholic potash solution, the addition being controlled by the indication of a drop of phenol phthalein in the solution. A slight excess of potash is used. After insuring saponification by heating, the solution is cooled, and then transferred to a 100 cc. cylinder by the use of ether, enough being used to bring the volume up to 100 cc. After thorough mixture by agitation, 1 gm. pure pulverized  $\text{Ag NO}_3$  is added, and the mixture again well agitated for 10 or 15 minutes. The silver resinate goes into solution in the ether. An

Aliquot portion of the clear ether solution is then siphoned off into another cylinder, and agitated with 20cc. of HCl. (1:2 by vol.) to set the rosin free. On evaporating a measured amount of the last ether solution the rosin is left behind, together with a little oleic acid (0.00235 gm. to every 10 cc. of ether solution taken.)

*Ulex* tests for FOREIGN SEEDS IN RAPE-SEED CAKE (*Repert. Anal. Chem.*, 1882, p. 3,) by distilling with water, and testing for sulphur in the distillate by oxidation with bromine and precipitation with barium salt, 233 parts Ba SO<sub>4</sub> = 99 parts oil of mustard. Rape-seed cake contains only 0.0238 % of that oil.

ALKALOIDS.—A solution of 1 gm. melted zinc chloride in 30 cc. of water is proposed by *Czumpelitz* (*Monit. Sci.* Feb. 1882). The substance to be examined is first dried, then moistened with the reagent and dried again. The colors produced are:

Strychnine,	Scarlet,	Quinine,	Pale Yellow,
Thebaine,	Yellow,	Digitaline,	Maroon,
Narceine,	Olive green,	Salicine,	Violet red,
Delphinine,	Red brown,	Santonine,	Violet blue,
Berberine,	Yellow,	Cubebine,	Purple,
Veratrine,	Red.		

When both strychnine and brucine are present the color obtained is a dirty yellow.

*R. C. Woodcock* (*Chem. News* XLV, 79) proposes TO SEPARATE ALKALOIDS from articles of food, &c., by the use of cubes of jelly prepared from a 6 % solution of gelatine. (Applied by *Dupre Analyst*, I, 186, to separate certain adulterants of wines.) In an experiment with strychnine, one part was satisfactorily separated from a million parts of substance.

*H. Beckhurst* (*Chem. Centr. bl.* XIII, 258) finds that the reduction of ferric compounds is not characteristic for the PTOMAINES. To extract alkaloids from CINCHONA BARK *J. E. De Vrij*, (*Jour. Pharm. Chim.*, May, 1882) recommends Prollius's mixture consisting of :

Ether.....	88 parts.
Ammonia.....	4 parts.
Alcohol (92 to 96 %). ....	8 parts.

For the extraction and determination of the alkaloids in the above barks, methods are also given by *M. Fluckiger* (*Jour. Pharm. Chim.* Feb. 1882) and *E. R. Squibb* (*Ephemeris* I, 76 and 105). The first named boils the powdered bark with four times its weight of water



and then adds milk of lime, after which the mixture is evaporated to dryness. The dry mass is exhausted with ether, and after the addition of a little hydrochloric acid (36 cc. of  $\frac{1}{10}$  normal HCl. to the extract from 20 gms. of bark), the ether is distilled off. The solution is then acidified with HCl. and filtered. The cold solution is partly neutralized by  $\frac{1}{10}$  normal NaHO solution 40 cc., allowed to stand until it clears, decanted or filtered if necessary, and soda lye (of sp. gr. 1.3) added to complete precipitation of the bases. The precipitate is dried between filter paper, then over sulphuric acid, and finally in an air bath at 100° C. before weighing. To separate the quinine the precipitate is boiled with thirty times its weight of water for an hour, and filtered boiling. On cooling, the quinine hydrate is deposited. If pure, the dry alkaloid is soluble in twenty times its weight of ether. *E. R. Squibb* takes 1.25 gm. quick lime, adds 30 cc. hot water, and as soon as it is slaked stirs in 5 gms. pulverized bark, and allows it to digest for some time. After drying over a water bath, the powder is digested in a closed flask over a water bath with 25 cc. amylic alcohol for four hours; 60 cc. of ether are then added and the digestion continued for an hour more with frequent shaking. It is filtered and washed with a mixture of 10 vols. amylic alcohol to 40 of ether. The ether is then boiled off, and the residue transferred to a small flask by the use of amylic alcohol. The solution and washings should not be over 10 cc. Then 6 cc. water and 4 cc. normal oxalic acid solution are added, and the mixture shaken. The aqueous solution is then filtered off and the amylic alcohol washed repeatedly with diluted oxalic acid solution. The solution is evaporated to about 15 cc., 20 cc. pure chloroform added, and a slight excess of normal soda solution. The alkaloids are thus separated, and are taken up by the chloroform, which, after washing, may be evaporated, leaving the alkaloids pure.

By shaking the alkaloids thus obtained with 5 gms. ground glass and 5 cc. ether, and pouring the mixture upon a filter wetted with ether, rinsing the adherent particles on to the filter until 10 cc. have passed through, and then washing with ether until a second 10 cc. (caught separately) have passed through, and evaporating these portions of ether separately, a basis for the estimation of the quinia is obtained. The first 10 cc. contains quinia plus some other alkaloids, the second 10 cc. approximately the same amount of alkaloids, not quinia, as the first. The difference between the weights of the residues left is therefore quinia.

*E. R. SQUIBB* also gives a process for the ASSAY OF OPIUM (*Ephe-*



meris I, 14), a modification of Fluckiger's process. Careful sampling and the determination of moisture are both indispensable. 10 gms. of the sample are first digested with 100 cc. water with frequent shaking for several hours. It is then filtered and percolated with water until the filtrate has a bulk of 120 cc. The residue is again shaken with more water and the percolation repeated, the filtrate being kept separate, and evaporated down to about 20 cc. before adding the stronger solution (of 120 cc.), the object being to avoid the effect of prolonged heat upon the major part of the alkaloids. The residue should be dried at 100° C. and weighed. The entire extract is finally evaporated to 20 cc.—cooled—5 cc. of alcohol added and well stirred in, then transferred to a tared flask, being mixed in with about 5 cc. water, 5 cc. more alcohol is added and after thorough admixture 40 cc. of ether. After vigorous shaking 4 cc. of ammonia (sp. gr. 0.96) is added, and the shaking is repeated until the crystals of morphia begin to separate, when the flask must be put aside for 12 hours. The ether solution is then decanted off through a filter wetted with ether, 20 cc. of ether is added to the flask and rinsed around without shaking and decanted through the filter. The funnel should be kept covered as much as possible while the ether is filtering through. Finally pour the aqueous solution on to the filter, rinse with a little water. Dry the filter and contents first with bibulous paper, afterward in the air bath (at 100°C) weigh, and deduct weight of filter. Invert the flask, dry and weigh it to get the weight of the morphia crystals which may adhere. The crystals of morphia are light brown or a loss has been sustained. 0.1 gm. of the precipitate when pulverized should dissolve completely on shaking in a corked test tube with 10 cc. of lime water (absence of narcotine). A description and criticism of the various methods for determining NICOTINE IN TOBACCO are given by *R. Kissling* (*Fres. Zeits. Anal. Chem.*, XXI. 64). The conclusion reached is that only Schloesing and Dragendorff's methods are at all satisfactory. The results of a number of experiments are detailed, and a list of analyses of 36 tobaccos given.

For the ESTIMATION OF TANNIN *F. Simaud* (*Dingl. Jour.*, CCXLIV., 391) proposes a modification of Lœwenthal's method. In extracting, paper saturated with a solution containing ferrous chloride and sodium acetate is used to determine when the extraction is complete. 10 cc. of the extract should not require over 12 cc. of the permanganate solution. The permanganate solution should be one gm. per litre, 1 cc.=0.00135 tannin, 20 cc. of the indigo solution

should be equivalent to 18 to 20 cc. of permanganate. 20 cc. of the solution is used for each determination. The gelatine solution is of the strength, &c., prescribed by Lœwenthal. To obtain satisfactory results close attention must be paid to the degree of dilution and the amount of acid used.

TO DETERMINE GLYCERINE in dilutions thereof, it is proposed, (*Jour. Soc. Chem. Ind.*, I, 75), to moisten 5 to 60 gms.  $\text{PbO}_2$  with 2 to 3 gms. of the dilution, and to dry the mixture for six hours in a desiccator, then one hour at  $100^\circ \text{C}$ . and finally one hour at  $120^\circ \text{C}$ . when it is cooled and weighed.  $\text{C}_2\text{H}_5\text{O}_2\text{Pb}$  forms. The gain in weight multiplied by 1.3429 gives the weight of glycerine actually present.

TO DETERMINE PHENOL *P. Diacosa* (*Repert. Anal. Chem.*, II, 137), proposes a reversal of the usual volumetric methods, viz., 20 cc. of bromine water the strength of which has been determined by the use of a standard solution of phenol, receives the addition of the solution containing phenol until the clear solution no longer acts upon iodized starch paper.

Jorissen's REACTION FOR FUSEL OIL.—A red coloration with anilin and  $\text{HCl}$ . is stated by *K. Forster* (*Ber. D. Chem. Ges.*, XV, 230), to be due to the presence of furfurol and not to that of amyl alcohol.

To determine ACETON IN WOOD SPIRIT *M. Kramer* (*Monit. Sci.*, Jan., 1882), adds 1 cc. of the sample, first 10 cc. of binormal solution of soda, then 5 cc. of binormal solution of iodine and agitates. The precipitate of iodoform is dissolved in 10 cc. of ether, and 5 cc. are evaporated on a weighed watch glass. From this the proportion of acetone is calculated. If the wood spirit is to be used in the manufacture of coal tar colors, not over 1 per cent. of acetone should be present.

TO TEST ASPHALTUM, *D. Claye* (*Jour. Soc. Chem. Ind.* I, 203), dissolves in  $\text{CS}_2$ , filters, and evaporates off the solvent. The residue is then pulverized, and 0.1 gm. is treated with 5 cc. fuming sulphuric acid for 24 hours, and then poured slowly into 10 cc. of water with continuous stirring. Pure asphalt gives a clear light yellow solution, while the adulterants—pitch, coal tar, etc.—give a dark color.

URINE EXAMINATIONS—FOR UREA, *J. R. Duggan* (*Am. Chem. Jour.*, IV, 47), obtains the best results by first mixing the urea and soda solutions and then adding the bromine. The results of *T. G. Wormley's* experiments with Apjohn's apparatus (*Chem. News*, XXI, 37), are given as follows (*Chem. News*, XLV, 27): To

insure the elimination of the whole of the nitrogen—1. The reagent must be freshly prepared. 2. The urea solution should be wholly added to the reagent, none of the latter being allowed to mix with the urea solution in the containing tube. 3. The amount of urea operated upon should not exceed one part to 1200 of the diluted reagent.

The volumetric uranium solution for PHOSPHORIC ACID cannot, according to *G. Guerin* (*Jour. Pharm. Chem.*, Feb., 1882), be successfully applied to urine examinations. According to this author, the uranium solution only gives accurate results when standardized in the presence of the same kind of salts as those existing in the substance to be analyzed.

List of books published on analysis during 1882 :

THE LABORATORY GUIDE: A Manual of Practical Chemistry, etc. By *A. H. Church*. Fifth Edition. London: J. Van Voorst.

TABLES FOR QUALITATIVE CHEMICAL ANALYSIS. *H. Will*. Third American Edition. Edited by *C. F. Himes*. Philadelphia : H. C. Baird & Co.

HANDBOOK OF VOLUMETRIC ANALYSIS. *F. Sutton*. Fourth Edition. London: J. and A. Churchill.

COMMERCIAL ORGANIC ANALYSIS. Vol. II. *A. H. Allen*. London : J. and A. Churchill.

QUANTITATIVE ANALYSE AUF ELEKTROLYTISCHEM WEGE. *Dr. Alexander Classen*. Aachen : J. A. Mayer.

ANALYSE VON PFLANZEN UND PFLANZEN-THEILEN. *Dr. Georg Dragendorff*. Gottingen : Vandenhoeck & Ruprecht.

FOODS : THEIR COMPOSITION AND ANALYSIS. *A. W. Blyth*. London : Charles Griffin & Co.

MANUAL OF SUGAR ANALYSIS. *J. H. Tucker*. New York : D. Van Nostrand.

HANDBOOK OF THE POLARISCOPE. From the German of Landolt. By *D. C. Robb* and *V. H. Veley*. London : MacMillan & Co.

## ABSTRACTS.

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Abstracts from the *Journal of Chemical Society*, London, by Arthur H. Elliott, Ph. B., F. C. S.

**On the Action of Aldehydes on Phenanthraquinone in Presence of Ammonia.** (Third Notice). BY FRANCIS R. JAPP, M. A., Ph. D., and FRED W. STREATFIELD. Vol. XL., p. 146.

This paper is a continuation of the work of the former of the two authors and Mr. Wilcock (*Chem. Soc. J. Trans.*, 1881, p. 228). The authors give an account of the product obtained by the action of salicylaldehyde upon phenanthroquinone. It has the composition  $C_{21}H_{14}N_2O$ ; crystallizes in very fine silky needles; fuses and decomposes at  $270-276^{\circ}C$ .; its best solvents are glacial acetic acid and amyl alcohol. Behaves like a phenol with caustic alkalies forming compounds. It has the properties of a weak base also. Fusion with potash gives salicylic acid.

A benzoyl derivative of the above compound was formed, which consisted of crystals of two forms, cubes and needles, fusing at  $218-220^{\circ}C$ . Analysis led to the formula of a mono-benzoyl derivative  $C_{21}H_{13}(C_7H_5O)N_2O$ . Authors discuss the constitution of the original  $C_{21}H_{14}N_2O$ .

The action of parahydroxybenzaldehyde upon phenanthraquinone as above is also described. The product is obtained in fine white needles, not fusing within range of mercury thermometer. Soluble in glacial acetic acid and amyl alcohol, also in dilute caustic alkalies. Its formula is given as  $C_{21}H_{14}N_2O$ . An acetyl derivate of the preceding body was formed. It crystallizes in needles which fuse at  $205-210^{\circ}C$ . The formula is  $C_{21}H_{13}(C_2H_3O)N_2O$ ; it is very soluble in glacial acetic acid.

Phenanthraquinone as above was acted upon by orthomethoxybenzaldehyde. The purified product was yellow needles fusing at  $207-208.5^{\circ}C$ . The mother liquor gives white crystals, fusing at  $144.5-145.5^{\circ}C$ . The yellow product gave formula  $C_{22}H_{16}N_2O$ ; it is readily soluble in boiling benzine, amyl alcohol, and glacial acetic acid, much less soluble in the cold; it is insoluble in dilute acids and caustic alkalies. The white product gave the formula  $C_{22}H_{15}NO_2$ ; it is readily soluble in hot benzine, amyl alcohol and glacial acetic acid; insoluble in dilute acids and caustic alkalies.

**Application of the Aldehyde and Ammonia Reaction in Determining the Constitution of Quinones.** BY FRANCIS R. JAPP, M.A., Ph.D., and FRED. W. STREATFIELD. Vol. XL., p. 157.

Authors apply the reaction to chryso-quinone and beta-naphtha-quinone. The chryso compound was obtained in silky needles, fusing at 259–265° C., and of a slight yellow tinge. Analysis gave  $C_{16}H_{10}NO$  as formula. Sublimation gave a white product. The new body is *benzenylamidochrysole*.

The experiment with beta-naphtha-quinone gave negative results.

**On the Action of Sodium Hydrate and Carbonate on Felspars and Wollastonite.** BY WALTER FLIGHT, D.Sc., F.G.S. Vol. XL, p. 159.

Author experimented upon adularia from St. Gothard, microlin from Siberia, albite from the Dauphiné, and a specimen of wollastonite from Sweden. These were heated in fine powder with solutions of sodium hydrate of different strengths, and also with solution of sodic carbonate. In the case of sodium hydrate, microlin lost 14.693 per cent. in strong solution and 7.453 in weak solution. Albite, under the same treatment, lost 23.1 per cent. and 9.472 per cent. respectively. The solutions contained both silica and alumina. With sodic carbonate solution albite lost 1.785 per cent.

In the case of wollastonite the sodium hydrate acts as a solvent of the whole mineral.

**On Pentathionic Acid. (Part II.)** BY WATSON SMITH and T. TAKAMATKE. Vol. XL, p. 162.

This paper opens with a defence of the criticisms of V. Lewes' Spring and Th. Curtius upon the authors' previous paper upon this topic. The authors give a number of details of experiments made to establish their position. They have proved "that the attempt to neutralize pentathionic acid with alkaline earth carbonates simply results in the formation of tetrathionates, with separation of sulphur." They further "conclude that on partial saturation of a pentathionic acid solution with an alkali, say potash, we may get a certain amount of potassium pentathionate formed, together with the realization of Stingl and Morawski's equation,  $5 H_2S_5O_6 + 10 KOH = 5 K_2S_4O_6 + 5 S + 10 H_2O$ .

**On Some Constituents of Resin Spirit.** By G. HARRIS MORRIS, F.C.S. Vol. XL, p. 167.

The product obtained from the lower fractions of resin spirit after standing some time with water in large flasks is the subject of this paper. The water solution yields upon evaporation colorless crystals of the composition  $C_7H_{14}O_2H_2O$ , and with a vapor density 63.6. The crystals are readily soluble in alcohol, ether, benzole. Heated they begin to sublime at  $100^\circ C.$ , giving off water. The dehydrated body melts at  $89.5$  and boils at  $195.6^\circ C.$  without decomposition. The water solution is neutral to test paper, and is not precipitated by basic lead acetate and ammonio-silver nitrate. The crystals warmed with hydrochloric acid and added to alcohol give a fine rose color, which is very characteristic.

The crystals are obtained in the largest quantity from the fraction boiling between  $100$ — $105^\circ C.$ , although the fractions from  $94$ — $150^\circ C.$  yield crystals.

A hydrocarbon from resin spirit boiling at  $103$ — $104^\circ C.$  was examined and found to be a heptene with a vapor density of 94.23. By the action of nitric acid a dinitro-heptylene was obtained having the formula  $C_7H_{12}(NO_2)_2$ ; it is easily soluble in alcohol, ether, and benzole, and insoluble in water. It melts at  $182^\circ C.$  The other products of the action of nitric acid upon heptene are carbonic dioxide, formic, acetic, butyric, and succinic acids.

With potassic dichromate heptene gives carbonic, dioxide, and acetic acids. With potassic permanganate it gives an acid not yet examined, and no carbonate.

The crystals mentioned above give with nitric acid the same products as the hydrocarbon, except formic acid. The dehydrated crystals are considered to be methylpropylallyleneglycol. These gave the diacetate  $C_7H_{12}(C_2H_3O_2)_2$ , which crystallizes in square plates, melting at  $68.5^\circ C.$ , and very soluble in alcohol, ether, and benzole.

A bromine compound  $C_7H_{12}Br_4$  was also formed from an aqueous solution of the crystals; and by passing gaseous hydriodic acid into the hydrocarbon,  $C_7H_{12}I$  or moniodide was formed.

**On the Preparation of Diethylnaphthylamine.** By BERNARD E. SMITH. Vol. XL, p. 180.

This body is best prepared by heating 10 parts of naphthylamine and 15 parts of ethylbromide, with a small quantity of ethyl alcohol to dissolve the naphthylamine, in a sealed tube for 8 hours, at  $120^\circ$

C. After purification, diethylnaphthylamine is a pale straw-colored oil, boiling at  $290^{\circ}\text{C}$ ., and darkening on exposure to light and air. Analysis gave carbon 84.18 and 84.25, hydrogen 8.78 and 8.97, nitrogen 7.25 and 7.33 per cent.

Diethylnaphthylamine hydrochloride is formed by adding hydrochloric acid to the base. The double platinum salt has the formula  $(\text{C}_{10}\text{H}_7(\text{C}_2\text{H}_5)_2\text{N} \cdot \text{HCl})_2 \text{Pt Cl}_2$ . Nitroso-diethylnaphthylamine is formed by adding sodic nitrite to a glacial acetic acid solution of the base. It crystallizes in reddish-golden scales, soluble in ether, alcohol, and benzine, and melting at  $165^{\circ}\text{C}$ .

**On the Action of Sulphuric Acid Upon Diethylnaphthylamine at High Temperatures.** BY BERNARD E. SMITH, Vol. XL., p. 182.

Author heated 20 grms of the base with 20 grms of sulphuric acid for eight hours, at  $190\text{--}210^{\circ}\text{C}$ . After adding excess of barium hydrate, and decomposing the barium salt with hydrochloric acid, the solution was mixed with excess of ammonia, which gave a white flocculent precipitate. This precipitate, crystallized from alcohol, gave colorless needles, blackened by light and air. This body melts at  $190^{\circ}\text{C}$ , and distills at above  $360^{\circ}\text{C}$  without change. Analysis gives  $(\text{C}_2\text{H}_5)_2\text{N} \cdot \text{C}_{10}\text{H}_7 \cdot \text{C}_{10}\text{H}_7\text{N}(\text{C}_2\text{H}_5)_2$  as the formula. It is very soluble in hot alcohol, not very soluble in ether, but readily so in benzine and chloroform. The hydrochloride is readily formed from the base.

The solution from the barium precipitate above mentioned, gave an acid with the formula,  $\text{HOO} \cdot \text{SC}_{10}\text{H}_7\text{N}(\text{C}_2\text{H}_5)_2 \cdot \text{SO}_3\text{OH}$ .

**On the Action of Carbon Oxydichloride (*Phosgene gas*) upon Diethylnaphthylamine.** BY BERNARD E. SMITH. Vol. XL., p. 185.

The base was dissolved in dry benzine, and the solution saturated with phosgene gas; a white crystalline mass separated out, which, after evaporating the benzine, was treated with hot water, and dilute hydrochloric acid. The latter only dissolved out the hydrochloride of the original base. The insoluble residue was dissolved in alcohol and crystallized. Fractional distillation gave three distinct bodies: 1. Colorless, transparent body, crystallizing in quadratic prisms, melting at  $70^{\circ}\text{C}$ ; with the formula  $\text{COCl} \cdot \text{C}_{10}\text{H}_7\text{N}(\text{C}_2\text{H}_5)_2$ . 2. A colorless, crystalline body, crystallizing in hexagonal prisms, melting at  $225^{\circ}\text{C}$ , and isomeric with the first body. 3. A colorless



crystalline body, crystallizing in rhombs with high refracting power, and melting at  $130^{\circ}\text{C}$ , and containing no chlorine.

By the action of the bodies containing chlorine upon the original base, a compound like the third body  $\text{C}_{14}\text{H}_{11}\text{N}_3\text{O}_2$  was obtained.

**Contributions to the Chemical History of the Aromatic Derivations of Methane.** By RAPHAEL MELDOLA. Vol. XL., p. 187.

Author gives the results of the action of benzyl chloride upon diphenylamine. He concludes that the crude products of the action are derivations of triphenylmethane and diphenylmethane. An account of the preparation of diphenylamine green on a large scale is given, and the formation of an alkaline salt of its sulphonic acid. The crude product was called "Viridine," and the salt of the sulphonic acid an "alkaline green." The constitutional formula of the hydrochloride is given, and the empirical  $\text{C}_{21}\text{H}_{18}\text{N}_2\text{Cl}$ . The formula of the base was found to be  $\text{C}_{21}\text{H}_{18}\text{N}_2\text{O}$ . Author mentions other methods of obtaining the green coloring matter; and compares its absorption spectrum with the absorption spectra of methyl and malachite green. In speaking of the sulphonic acids of diphenylamine, the author mentions two of them, a mono- and a di-sulphonic acid. The chloride separates from the alcoholic solution, containing hydrochloric acid, in a granular state; and the base separates from the alcoholic solution, on adding ammonia, as a gummy mass which becomes solid.

Of the derivations of diphenylmethane formed simultaneously with diphenylamine green, the author mentions a white amorphous powder, melting about  $89^{\circ}\text{C}$ ., and giving no color upon oxidation. This proved to be phenylamidodiphenylmethane  $\text{C}_{19}\text{H}_{17}\text{N}$ . It is soluble in benzine, chloroform, ether and carbon disulphide; insoluble in glacial acetic acid, alcohol and acetone.

By heating diphenylamine with two molecules of benzyl chloride and zinc chloride, the author obtained benzyl-phenylamido-diphenylmethane  $\text{C}_{26}\text{H}_{23}\text{N}$ .

By the action of benzyl chloride upon aniline, the author dibenzylamido-diphenyl-methane  $\text{C}_{27}\text{H}_{25}\text{N}$ , which resembles the above-mentioned bodies in appearance, is less soluble in benzine and toluene, and its solutions give blue fluorescence.

The preceding reaction repeated with acetanilide shewed replacement of the acetyl group by benzyl. All the diphenylmethane derivations in this paper are white amorphous powders. The paper is



full of the most interesting theoretical considerations and constitutional formulæ.

### **Contributions to the Chemistry of Cerium Compounds.**

By W. N. HARTLEY, F. R. S. E. No. XL., p. 202.

Author prepared pure cerium salts by two methods, that of Mosander and that of Bunsen and Bahr. The purity of the salts was determined by comparing photographs of spectra of 10 per cent. solutions of cerium, didymium, and lanthanum, on the same plate.

A very delicate test for cerium is given thus: To the solution to be examined, either neutral or slightly acid, add ammonia acetate and a little hydrogec peroxide. Much cerium if present gives a brown color, and shaking precipitates gelatinous ceric acetate. With a small quantity of cerium the liquid turns yellow without a precipitate forming. By this method one part of cerium can be separated from 100,000 parts of liquid.

The preparation of cerous phosphate is best performed by adding sulphurous acid to the cerous solution, and then adding an acidified solution of sodium phosphate; gently warming causes the precipitate to settle rapidly. The precipitate is white, and dries to a porcelain-like mass, very hard to pulverize. Author makes the composition  $\text{CePO}_4, 2 \text{H}_2\text{O}$ .

Ceric phosphate was prepared from solutions of ceric nitrate and sulphate, and although the conditions were varied the composition was the same, and the author concludes that there is but one ceric ortho-phosphate  $(\text{Ce O}_2)_4 (\text{P}_2\text{O}_5)_3$ .

### **The Analysis of Rhabdophane, a New British Mineral.**

By W. H. HARTLEY, F. R. S. E. No. XL., p. 210.

The mineral has the general composition  $\text{R}_2\text{O}_3, \text{P}_2\text{O}_5, 2 \text{H}_2\text{O}$ , in which Ce, Di, La, or Yt may wholly or in part replace each other. Author's analysis gave water (loss on ignition) 9.34—silica 0.36— $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ , with  $\text{P}_2\text{O}_5$  0.21—magnesium phosphate 1.09—cerous oxide 23.19—lanthanum and didymium oxides 34.77—yttrium oxide 2.09—phosphoric anhydride 24.77 per cent. Author gives the results of a most careful search for yttrium, with tables of the lines obtained in the spectrum, using a Rutherford's grating with 17,460 lines to the inch. There was a complete absence of lines attributable to erbium; but there were four lines in green which the author could not connect with any known element.

This is the first instance on record of yttrium being found in any British mineral.

**On Benzyl-Phenol and its Derivations.** (*Part II.*) By EDWARD H. RENNIE, M.A., B.Sc. No. XL., p. 220.

Author describes the following bodies : *Benzyl-phenol—sulphonic acid* ; not obtained in a pure state as it loses weight at 100° C and partially decomposes. *Mono-nitro-benzyl-phenol*, golden yellow prisms, melting at 74-75 C°, volatile in steam. The potassium derivative, in needles, have a brick-red color. *Amido-benzyl-phenol*; the hydrochloride forms glistening scales, which are unstable. *Dinitro-benzyl-phenol* ; crystallized from alcohol; it melts at 87-88° C. The potassium derivative crystallizes in orange needles and yields benzoic acid with chromic liquor. *Para-nitro-benzoic acid* was obtained by oxidizing trinitro-benzyl-phenol with chromic liquor ; it melts at 238° C. *Nitro-bromo-benzyl-phenol* prepared from potassium bromo-benzyl-phenol-sulphonate and crystallized from alcohol, gives brilliant yellow scales, melting at 64-65° C. The potassium derivative crystallizes in brilliant red scales. The above sulphonate may also be prepared from nitro-benzyl-phenol-sulphonate by treating with bromine in a glacial acetic acid solution. Several other ways of preparing this body are also given. By acting on the above-mentioned nitro-bromo compound with nitric acid, *Dinitrobromphenol* was obtained, which melts at 118° C., and crystallizes from alcohol in fine, pale yellow needles.

In discussing the constitution of benzyl-phenol and its derivations the author gives the results of experiments upon the oxidization of benzyl-phenol-methyl ether. From this ether he obtained *Methoxybenzophenone* in four-sided prisms, melting at 61-62° C. From paraoxybenzophenone and methyl iodide he obtain the same body, and hence concludes that it is paramethoxybenzophenone, and that it proves that benzyl-phenol is a para-derivative.

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Abstracts from *Berichte der Deutschen Chemischen Gesellschaft*, by L. H. Friedburg.

**Bromo-substitution Products of Sebacylic Acid.** FR. GANTTER and CARL HELL. (Vol. XV., p. 142.) It seems, according to these researches, that red phosphorus acts "catalytically." between bromine and sebacylic acid.

**Synthesis of the Homologous Phenols.** AD. LIEBMANN (Vol. XV., p. 150.) Preparation of isobutyl-phenol, amyl-phenol, butyl-phenol, and benzyl-phenol.

**Determination of Sulphurous Acid in Wine.** B. HAAS. (Vol. XV., p. 154.)

**Lecture Experiments.** MAX. ROSENFELD. (Vol. XV., p. 160.) Use of glass tubes for showing the decomposition of water by red-hot iron.

Decomposition of steam by magnesium.

Combustion of ammonia in oxygen.

**On the Etherification of the Oxyacids.** N. MENSCHUTKIN. (Vol. XV., p. 162.)

**A Synthesis of Thymol out of Cuminol.** OSCAR WIDMANN. (Vol. XV., p. 166.) Cuminol is transformed into nitro-cuminol, the latter treated with phosphorus pentachloride, is changed into nitro-cymylen-chloride; zinc, and sulphuric acid as reducing agents, hydrogenizing the nitro-chloro compound into cymidin. Nitrite of potash then transforms the cymidin into nitrosothymol, which was proved to be identical with nitrosothymol, prepared from thymol and nitrous acid in the same manner as previously indicated.

This is the first synthesis of a derivative of thymol, hence of thymol.

**Preparation of Succinic Acid from Tartaric Acid by Fermentation.** F. KONIG. (Vol. XV., p. 172.) The fermenting liquid is a solution of tartrate of ammonia. Acetic and formic acids are formed in varying quantities, also carbonate of ammonia. The process is recommended as a good one for making succinic acid, even on a large scale.

**Preparation of Coloring Matter by the Reaction of Aromatic Nitro-substances on Phenols and on Poly-atomic Alcohols in Presence of De-hydrating Agents.** HEINRICH BRUNNER. (Vol. XV., p. 174.) A representative of the first-mentioned series is prepared by reaction between resorcin and nitrobenzol. Of the second class two combinations have been formed, respectively from erythrit and nitro-alizarine, and cane sugar and nitro-alizarine.

**Oxy-propyl-toluidin.** R. F. MORLEY. (Vol. XV., p. 179.)

**Mono-phenyl-boro-chloride and some of its Derivatives.** A. MICHAELIS and P. BECKER. (Vol. XV., p. 180.) The mono-phenyl-boric acid has only a mild toxic influence upon the human system, while it is comparatively poisonous for lower organisms, bacteria, &c. The free acid is about ten times stronger as an anti-septic than its sodium salt.

**OnTolyl-Methyl-Keton.** A. MICHAELIS. (Vol. XV., p. 185).

**Reaction between the additional products of Chinolin plus halogen-alkalies and oxide of silver.** W. LACOSTE. (Vol. XV., p. 186).

Isolation of basic compounds, corresponding to a quarternary oxide of ammonium, by means of the above named reaction.

**Reaction of Dimethyl-phenyl-phosphine upon Bromide of Ethylene.** L. GLEICHMANN, (Vol. XV., p. 198).

**Researches on the Laws of substitution in the Naphthalene series.** HENRY E. ARMSTRONG. (Vol. XV., p. 200).

**Formation of basic compounds from acido-amides.** O. WALLACH. (Vol. XV., p. 208).

**Influence of heat upon Thio-formanilide.** W. W. I. NICHOL. (Vol. XV., p. 211).

**Syntheses by means of Phenyl-acetylene and its derivatives.** ADOLF BAEYER and LUDWIG LANDSBERG. (Vol., XV., p. 212).

**On the question of identity between the coloring matter of the Sophora Japonica Capparis Spinosa and Ruta Graveolens, with Quercitrin and Quercetin.** P. FOERSTER. (Vol. XV., p. 214).

**Decomposition of mono-halogenized crotonic acids by alkalies.** R. FRIEDRICH. (Vol. XV., p. 218).

**Analyses of some Nephrites of Lake Dwellings.** K. SEUBERT and G. LINK. (Vol. XV., p. 219).

The proportions of silicic acid to bases and to water lead to the general formula  $R^{11} Si O_3$  of the group of hornblende. They, therefore, belong to the group of veritable nephrit (Cacholong of *Rammelsberg*): occurrence: Maurach on the Bodensee.

**On rendering oxygen active.** MORITZ TRAUBE. (Vol. XV., p. 222).

Palladium-hydrogen shaken with water and oxygen (air) yields immediately and abundantly peroxide of hydrogen. The oxidizing effects of palladium-hydrogen in presence of oxygen and water are not directly caused by the former, but almost exclusively by the peroxide of hydrogen generated. Potassium iodide starch mixture is excepted. Peroxide of hydrogen does not turn this blue, while palladium-hydrogen and oxygen do. Here palladium transfers the oxygen from peroxide of hydrogen to the iodide of potassium. Nascent hydrogen is not able to render oxygen active by splitting its molecules.

**On Anthramin.** C. LIEBERMANN and A. BOLLERT. (Vol. XV., p. 226).

Anthramin is Liebermann's name for the same compound, which Roemer calls anthracylamin= $C_{14}H_9NH_2$ , and which has been found simultaneously by both gentlemen, independent of each other, in the same laboratory. Anthramin is prepared from anthrol by amidizing it with acetamid in a closed tube at  $+280^\circ C.$ , and subsequent distillation of the products of the reaction with potassium hydrate and water. Yield: Twenty per cent of the anthrol.

**Cause of Jorissen's reaction for Fusel Oil.** K. FOERSTER. (Vol. XV., p. 230).

Fusel oil, heated with aniline and hydrochloric acid, produces a beautiful red coloration of the mixture. On this fact Jorissen has founded a method for testing common alcohol for fusel oil (produced by fermentation).

Foerster's investigation shows that this coloration is due to an impurity, and that neither amylic alcohol nor its homologues cause the reaction.

The impurity was found to be furfural, of which the property of producing color-reactions with bases of the aromatic series is well known.

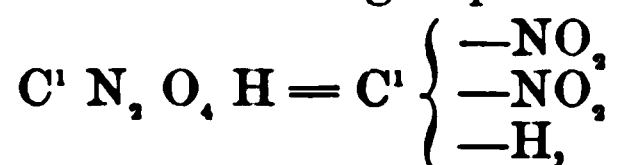
**The combinations of Benzo-tri-chloride with aromatic Bases.** OSCAR DOEBNER. (Vol. XV., p. 232).—The author published, some time ago, his researches on the action of benzotrichloride and phenols, as well as on tertiary aromatic bases, now he deals with the primary and secondary aromatic bases, beginning with aniline. New dyeing substances are among the results of this investigation

Abstracts from the *Comptes Rendus*, by A. Bourgougnon.

**Double Salts formed by the Haloid Salts of Mercury.**  
By M. BERTHELOT. (No. 7, Feb. 13th, 1882).

**Researches upon the Nitrogenized Acids derived from the Acetones.** BY G. CHANCEL. (No. 7, Feb. 13th, 1882).

By the action of nitric acid upon propion and upon butyron, ethylnitrous acid,  $\text{CH}_3\text{—C N, O, H}$ , and propylnitrous acid,  $\text{C}_2\text{H}_5\text{—CN, O, H}$ , are formed. These two compounds are identical with dinitro-ethan and dinitro-propan. The characteristics of all these nitrogenized acids is the univalent group



of which the saturation is completed by one atom of hydrogen (methylnitrous acid) or by an alcoholic radical; the general formula of alkylnitrous acids is then



By the action of reducing agents the group  $\text{C N, O, H}$  is transformed into ammonia, hydronylamin and  $\text{C O O H}$ , characteristics of the organic acids.

The formation of alkylnitrous acids by the action of nitric acid upon the acetones is represented as follows :



*Amylnitrous acid*,  $\text{C}_5 \text{ H}_{11}\text{—CN, O, H}$ . Pentyl or amylnitrous acid is obtained by the reaction of nitric acid upon normal capron.

*Amylnitrite of Potassium*,  $\text{C}_5 \text{ H}_{11}\text{—CN, O, K}$ ; yellow needles of fatty appearance.

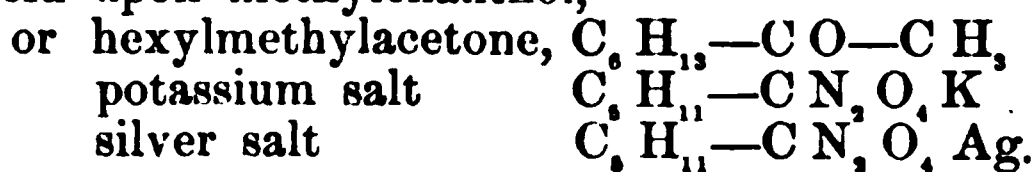
*Amylnitrate of Silver*,  $\text{C}_5 \text{ H}_{11}\text{—CN, O, Ag}$  is like the potassium salt, but less soluble.

*Butylnitrous acid*,  $\text{C}_4 \text{ H}_9\text{—CN, O, H}$ . Obtained by the action of nitric acid upon the most volatile part of crude capron, distilling at  $175^\circ \text{C}$ . Uncolored liquid, heavier than water, transformed into normal butyric acid by reduction.

*Butylnitrite of Potassium*,  $\text{C}_4 \text{ H}_9\text{—C N, O, K}$ . Yellow prisms.

*Butylnitrite of Silver*,  $\text{C}_4 \text{ H}_9\text{—C N, O, Ag}$ . Crystallizes with difficulty from a warm saturated solution.

*Herylnitrous acid*,  $\text{C}_6 \text{ H}_{11}\text{—C N, O, H}$ . Obtained by the action of nitric acid upon methylenanthol,



# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Room 1, University Building, N. Y.*

The last regular meeting was held Friday, October 6, 1882. The meeting was called to order at 8.45 P. M., Mr. P. Casamajor in the chair.

The minutes of the previous meeting were read, but as there was not a quorum present, they could not be acted upon.

After which, the first paper of the evening, "On the fractional dehydration of Ammonium Alum and the Atomicity of Aluminium," by Prof. J. W. Mallet, was read.

This paper describes a large number of interesting experiments, which go to show that Aluminium is either a triad or pentad.

After some remarks, the second paper "On the determination of Sulphur in Gas," by Mr. A. P. Hallock, was read.

Mr. Hallock stated that he used dehydrated sulphate of copper for the estimation of  $H_2S$ .

The apparatus used, consisted in a series of U tubes; the gas being allowed to pass through them for a certain length of time.

Dr. Grothe then inquired whether the above gas contained any Ammonia? Mr. Hallock stated that it did, but only in traces.

Dr. Grothe further asked whether the above gas contained any acetylene? Mr. Hallock replied that there was none. Upon which Mr. Casamajor remarked that possibly sheet rubber would remove small traces of hydrocarbons.

Mr. Elliott then stated that the Municipal Gas contains no other hydrocarbons than  $CH_4$ , and that this is not absorbed by the dehydrated sulphate of copper.

He further stated that all the sulphur in water gas is in the shape of sulphuretted hydrogen.

Some remarks about the combustion of sulphur in gas, lead to a lively discussion, in which Mr. Elliott stated that the  $H_2S$  was all burnt to  $H_2SO_4$ , while Dr. Grothe and Mr. Casamajor insisted that  $SO_2$  is likewise formed.

The following gentlemen were then nominated :

Prof. Cuthbert P. Conrad, regular member. Proposed by J. W. Mallet, E. Waller and James H. Stebbins, Jr.

Ervin von Wilmouski, regular member. Proposed by James H. Stebbins, Jr., Wm. Rupp and Dr. Grothe.

Alfred L. Beebe, regular member. Proposed by J. B. McIntosh, A. L. Colby and A. H. Elliott.

John Cawley, regular member. Proposed by James H. Stebbins, Jr., A. H. Elliott and P. Casamajor.

N. Hathaway, regular member. Proposed by E. Waller, Wm. Rupp and C. E. Munsell.

Geo. Leguin, associate member. Proposed by Nelson H. Darton, James H. Stebbins, Jr., and Wm. Rupp.

John G. Macfarlan, associate member. Proposed by P. Casamajor, James H. Stebbins, Jr., and A. H. Elliott.

Dr. Waller then stated on behalf of the committee on papers, that the lack of MS. produced much trouble, and he invited the members to assist said committee, by supplying as much MS. or abstracts as possible.

There being no further business the meeting adjourned.

JAMES H. STEBBINS, JR.,

*Recording Secretary.*

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### Laboratory Notes.—By E. WALLER.

#### PRECIPITATION OF BARIUM SULPHATE.

I have frequently observed that solutions of barium chloride seem to dissolve more air or carbon dioxide than most other solutions used as reagents, so much so that the addition of a cold barium solution to a boiling solution causes a brisk evolution of gas comparable with the effervescence due to the addition of acid to a carbonate. When a sulphate is present in the solution to which cold barium chloride solution has been added, even though the sulphate solution may have been boiling, the precipitate separates in a very finely divided condition, which gives much trouble in filtering, as the precipitate is so fine as to pass through the pores of a filter, and takes very long to settle.

If the barium solution is boiled for a few minutes before adding it, this phenomenon does not occur. The conclusion seems to be that the gases dissolved by the barium solution play an important part in the matter of the separation of the precipitate. Other chemists have evidently experienced the same trouble, for I have found recommendations in the literature to add a pinch of starch to the solution, and also more recently the addition of a little silver



nitrate, the Ag Cl. being subsequently dissolved out by ammonia, both having the same object in view,—to cause the barium sulphate to separate promptly in a form suitable for filtration and washing.

However, by boiling *both* barium and sulphate solutions for a few minutes *separately* and mixing them when boiling hot, then boiling the mixture for a few minutes and finally allowing it to stand on the water-bath, the precipitate will settle completely in about half an hour.

#### PECULIAR REACTION WITH LEAD ACETATE.

Many of the members of the Society will remember a conversation of the Society which was held at the laboratory of our late fellow member, Dr. Lipps. Among other interesting things shown us was the preparation of Dr. Lipps' "dextrin reagent," where litharge was added to solution of lead acetate, and the mixture heated to about 55°C., when it thickened up, owing, no doubt, to the formation of some of the extremely basic lead compounds. The reaction is such a peculiar one that I have thought it worthy to be placed on record, which Dr. Lipps failed to do during his lifetime.

To obtain the reaction, take a cold saturated solution of lead acetate, warm to 50 or 60°C., and add litharge little by little, stirring it in vigorously. At a certain point the mixture suddenly thickens, and in a few minutes becomes so solid that the vessel may be inverted without spilling. After standing for a short time the mass may be extracted with water, and the solution constitutes the "dextrin reagent." This reagent, when boiled with solutions containing dextrin, gives a white precipitate. According to Dr. Lipps 100 parts of this precipitate, when thoroughly dried, corresponds to 35 parts of dextrin.

The compound formed is probably the sexbasic lead acetate described by Berzelius (Ann. Chim., XCIV., 296), containing—

Acetic acid.....	5.7 per cent.
Oxide of lead.....	91.3    “
Water.....	3.0    “

In Berzelius' description of the preparation of this acetate, ammonia was used to withdraw a portion of the acid from its combination with the lead. In Allen's Commercial Handbook, p. 318, the use of lead acetate to which ammonia has been added is mentioned as a reagent for dextrin. The compound formed is probably the same.

**Laboratory Notes.**—By JAMES H. STEBBINS, JR., S. B., F. C. S.

ANALYSIS OF GALENA FROM NEWBURYPORT, MASS.:

This ore was said to be rich in silver ; however, upon analysis, only a trace could be found, while the percentage of PbS is quite large.

The composition of this ore may be seen from the following figures :

Si O <sub>2</sub> .....	0.40
PbS.....	98.80
Sb, S <sub>2</sub> .....	0.40
FeS.....	0.15
Ag. trace.....	....
<hr/>	
Total.....	99.75

Its appearances closely resembled ordinary galena, having a cubic cleavage and coarse structure. Hardness about 2.5 ; lustre, metallic ; and streak, lead gray.

ANALYSIS OF PYRRHOTITE FROM CLAYTON, N. Y.:

This ore was handed to me to be tested for gold, but, upon analysis, no gold was revealed.

Its appearance was massive and amorphous. Hardness, about 3.5—4 ; lustre, metallic ; color, between bronze-yellow and copper-red. Several of its sides were tarnished. Streak, dark grayish black. It was, moreover, strongly magnetic when reduced to a fine powder.

Composition.

Si O <sub>2</sub> .....	3.30
Fe.....	58.03
S.....	38.26
P, O <sub>2</sub> trace.....	....
<hr/>	
Total.....	99.59

According to theory, it should have the following composition :

*Calculated for Fe<sub>7</sub>S<sub>8</sub>.*

Sulphur.....	39.5
Iron.....	60.5
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Total.....	100.0

It will, therefore, be observed that the above ore deviates but slightly from theory.

## ANALYSES OF THE CALCULI IN THE "WOOD" MUSEUM OF BELLEVUE HOSPITAL.

BY A. BOURGOUON.

In 1867 the late Dr. Jas. R. Wood deeded to the City of New York a most complete and valuable collection of anatomical specimens. Among other curiosities there were many calculi. Some of these deserve particular attention, from the fact that they have been removed by Dr. Wood; others because of their size or composition.

A classification of the specimens could not be made unless analysis had determined their composition, and with this view they were submitted to chemical tests.

As chemists, especially those interested in physiological chemistry, may not be aware of the existence of this Museum, the results of the analyses are submitted in a condensed form. The numbers attached to each specimen indicate those of the catalogue of the Museum.

970. Stone removed by Dr. J. R. Wood at his clinic at Bellevue Hospital, Jan. 10th, 1880. Several pieces, 13 grammes. A nucleus of wax and dried blood surrounded by layers of phosphate of ammonium and magnesium.

971. Weight, 70 grammes. Calcium oxalate, urate and phosphate.

972. 5 stones; weight, 94 grammes. Ammonio-magnesium phosphate, calcium urate.

973. Weight, 58.5 grammes. A nucleus of uric acid, ammonium urate, calcium urate and silica, surrounded by calcium phosphate and amm. magn. phosphate.

974. Weight, 63 grammes. Calcium urate, oxalate and phosphate.

975. Weight, 48 grammes. Calcium oxalate and urate.

976. Weight, 26.5 grammes. Calcium oxalate and phosphate.

977. Weight, 49.5 grammes. Mulberry calculus, well formed. Calcium oxalate and urate. The nucleus contains ammonium urate.

978. Weight, 34.5 grammes. Removed from a colored man at Bellevue Hospital. Uric acid, amm. magnesium phosphate, ammonium urate, calcium oxalate.

979. Weight 9 grms. A nucleus formed by a candle or lamp wick. Surrounded by uric acid, calcium urate and phosphate.
980. Weight 15 grms. Uric acid, calcium oxalate and urate.
981. Weight 11.5 grms. Mulberry calculus, calcium oxalate.
982. Weight 15 grms. Cholesterin (biliary.)
983. Weight 15 grms. Calcium oxalate, calcium phosphate.
984. Weight 22 grms. The patient was over 70 years of age. Amm. magnesium phosphate.
985. Weight 25.5 grms. Removed by Dr. J. R. Wood. Calcium oxalate and urate.
986. Weight 26 grms. Removed from a boy 13 years old, by Dr. J. R. Wood, Saturday, Nov. 3d, 1877, at Bellevue Hospital. Uric acid, calcium urate and oxalate.
987. Weight 29.5 grms. Only one-half of the calculus. Uric acid, calcium urate, and phosphate and silica.
988. Weight 21 grms. Removed Dec. 13th, 1877. The patient was 70 years old. Calcium phosphate.
989. Weight 36 grms. A nucleus of ammonium urate, surrounded by calcium phosphate, amm. magnesium phosphate.
990. Weight 8 grms. 6 stones removed from a boy 6 years old. Calcium phosphate and urate.
991. Weight 12 grms. Calcium oxalate.
992. Weight 6 grms. 7 stones removed from a boy 12 years old. Calcium phosphate and amm. magn. phosphate.
993. Several small pieces. Patient 22 years old. Calcium urate and oxalate.
994. Calcium urate and oxalate.
995. Uric acid, amm. magn. phosphate.
996. Weight 25 grms. Dried blood and calcium phosphate.
997. Weight 20 grms. Removed Nov. 15th, 1868. Patient 20 years old. Uric acid, calcium urate, amm. magn. phosphate.
998. Weight 20 grms. Removed Dec. 16, 1872. Uric acid, amm. magn. phosphate.
999. Weight 32 grms. Removed May 14, 1874. The patient was 64 years old. Amm. magn. phosphate.
1000. Weight 31 grms. Calcium urate and phosphate, and amm. magn. phosphate.
1001. Calcium oxalate and urate.

1002. Weight 20 grms. Patient 40 years old. Calcium phosphate and oxalate.
1003. Amm. magn. phosphate.
1004. Uric acid, amm. magn. phosphate, and calcium phosphate.
1005. Uric acid and amm. magn. phosphate.
1006. Uric acid, calcium urate and phosphate and silica.
1007. Amm. magn. phosphate calcium oxalate and phosphate.
1008. Amm. magn. phosphate and calcium phosphate.
1009. Calcium oxalate and phosphate.
1010. This is not a calculus, it is a water-worn stone, a kind of silex, which ought to be removed from the collection.
1011. Amm. magn. phosphate and calcium phosphate.
1012. Uric acid and calcium oxalate.
1013. Uric acid.
1014. Amm. magn phosphate, calcium urate, and uric acid.
1015. Uric acid, amm. magn. phosphate.
1016. Calcium urate, amm. magn. phosphate.
1017. Calcium urate and phosphate.
1018. Uric acid, calcium oxalate, amm. magn. phosphate.
1019. Calcium oxalate.
1020. Calcium oxalate and phosphate, and amm. and magn. phosphate.
1021. Calcium urate, phosphate, and amm., magn. phosphate.
1022. Calcium urate and amm. magn. phosphate.
1023. Two calculi, amm. magn. phosphate and calcium phosphate.
1024. Removed Dec. 13, 1870, at Bellevue Hospital. Calcium oxalate, amm. magn. phosphate.
1025. Removed from a child 2 years and 9 months old, by Dr. J. Wood. Uric acid and amm. magn. phosphate.
1026. Uric acid, calcium urate and silica.
1027. Uric acid, calcium phosphate.
1028. Calcium urate, amm. magn. phosphate.
1029. Amm. magn. phosphate, calcium phosphate and calcium urate.
1030. Calcium urate, amm. magn. phosphate.
1031. Uric acid.
1032. Amm. magn. phosphate, calcium oxalate.
1033. Amm. magn. phosphate.
1034. Removed Nov. 2d, 1872, by Dr. J. R. Wood, from a child 6 years old, at Bellevue Hospital. Calcium oxalate.
1035. Calcium oxalate.
1036. Two calculi, amm. magn. phosphate.
1037. Calcium oxalate, amm. magn. phosphate, calcium phosphate.

1038. Several small pieces removed from the bladder by lithotrity.

Uric acid, ammonium urate, amm. magn. phosphate.

1039. Several small pieces uric acid, amm. magn. phosphate.

1040. Uric acid.

1041. Cholesterin.

1042. Several pieces weighing 18 grms., removed by Dr. Ch. Phelps, May 9th, 1877, from a man 66 years old. Amm. magn. phosphate, calcium phosphate.

1043. Nucleus of a calculus removed by Dr. Ch. Phelps Nov. 30th, 1877. A pin surrounded by dried blood, uric acid and calcium phosphate.

1044. Several pieces calcium oxalate and phosphate.

1045. Renal calculus. Uric acid, amm. magn. phosphate.

1046. Renal calculus. Amm. magn. phosphates. Among the pieces of this calculus are three stones of cholesterin; they are here evidently by mistake.

1052. Debris from operation by lithotrity; patient 40 years old. Amm. magn. phosphate, calcium phosphate.

1056. Vesical calculus of horse, only one-half, weight 180 grms. Calcium carbonate (nucleus), amm, magn. phosphate.

Intestinal calculus of horse, of a spherical form, weighing 390 grms. (no number in the collection) has the following composition:

Organic matter.....	6.13	per cent.
Silica .....	0.24	"
Amm. magn. phosphate.....	86.24	"
Calcium carbonate.....	2.03	'
Magnesium.....	5.25	"
	<hr/>	
	100.00	

Before the analysis was made the powder of the calculus was dried at 100°C.

1057. Weight 25 gms. Cholesterin. Removed from (?) vagina of a woman 30 years old.

## ABSTRACTS.

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Abstracts from the *Journal of the Chemical Society*, London, by Arthur H. Elliott,  
Ph. B., F. C. S.

### On the Solubility of Glass in Certain Reagents. By RICHARD COWPER, A. R. S. M. (Vol. XL., p. 254.)

Author kept a measured quantity of each reagent in a sealed tube of hard glass for six days at 100°C. Two series of experiments, A. and B., gave the results shown in table below, on neutralizing with hydrochloric acid and evaporating to dryness and igniting in a platinum dish.

Reagent.	No. Mgrms. dissolved by 100 cc.	
	A.	B.
Water.....	8.0	10.0
H <sub>2</sub> S .....	12.5	8.7
(N H <sub>4</sub> ), S dilute, prepared from NH <sub>4</sub> OH of sp. gr. 0.982.....	49.6	52.5
(NH <sub>4</sub> ), S concentrated, prepared from NH <sub>4</sub> OH of sp. gr. 0.88.....	34.	47.2
NH <sub>4</sub> OH dilute, sp. gr. 0.982.....	25.8	42.5
NH <sub>4</sub> OH concentrated, sp. gr. 0.88.....	7.5	7.7
NH <sub>4</sub> HS, prepared from NH <sub>4</sub> OH sp. gr. 0.982.....	—	51.2

Another experiment in which dilute (NH<sub>4</sub>), S was boiled in a flask, 60 cc., gave 0.008 gram. residue.

A series of experiments to find the degree of dilution of Am OH for maximum effect gave following results:

40 cc., taken.				Dissolved in 100 cc.	
NH <sub>4</sub> OH	sp. gr. 0.88	+1 vol	H <sub>2</sub> O.....	22.5	
"	"	+2 vols	" .....	26.2	
"	"	+3	" .....	30.0	
"	"	+4	" .....	26.7	
"	"	+5	" .....	41.2	
"	"	+6	" .....	32.0	

### Analysis of a Piece of Oxidized Iron from the Condenser of H. M. S. "Spartan." (Vol. XL., p. 256.) By RICHARD COWPER, A. R., S. M.

The details of the methods of analysis are given, and the following results:

Insoluble Residue dried at 120° C	30.14	{ C.....12.57 H......24 Mineral .17.54	{ SiO <sub>2</sub> 16.98 Fe <sub>2</sub> O <sub>3</sub> .12 Al <sub>2</sub> O <sub>3</sub> .06 CaO .15 MgO .02
Cupric oxide.....	.38		
Ferric ".....	2.21		
Ferrous oxide.....	42.33		
Alumina.....	.16		
Manganic oxide.....	1.02		
Cobalt oxide.....	.05		
Sodium oxide.....	.11		
Phosphoric acid.....	5.24		
Sulphuric ".....	.31		
Vanadic ".....	.11		
Chlorine.....	2.08		
Water .....	16.71		

**Note on a Convenient Apparatus for the Liquefaction of Ammonia.** (Vol. XL, p. 259.) BY J. EMERSON REYNOLDS, M. D., F. R. S.

The apparatus consists of two vertical wrought iron tubes joined below by a horizontal tube of the same metal. The vertical tubes are 40 and 30 cm. high and 12 cm. internal diameter. The horizontal tube is 25 cm. long and 5 or 6 cm. internal diameter. The higher vertical tube has a gas-tight screw cap. The lower vertical tube also has a tight-fitting cap, which is perforated to receive the slender stem of a tube very much like a thermometer, which is cemented into this cap. The tube in the cap is about 25 c. m. long and the bulb below terminates in a beak which is always open. To fill the tube with dry ammonia gas, both ends are left open, and the gas allowed to flow through to expel all air. Now the upper end is sealed by a blow-pipe, and the lower beak dipped into mercury, which enters and prevents the escape of the gas.

The cap of the highest vertical iron tube is now removed and the iron apparatus mentioned above is filled with mercury until the lower tube is nearly full. The glass tube with the ammonia gas is now placed in the lower iron tube, and the cap which holds the glass tube is screwed tightly home, with a leather washer. From the higher



iron tube remove enough mercury to leave about 12 cm. below the surface of the cap ; fill this space with strongest liquor ammonia and screw the cap into its place. By heating carefully the higher iron tube containing the ammonia solution, a pressure is exerted upon the glass apparatus in the lower tube and the ammonia gas is liquified. This apparatus may be used for other gases.

**Transformation of Urea into Cyanamide.** (Vol. XL. p. 262)  
By H. J. H. FENTON, M.A.

Author dehydrates urea by heating with metallic sodium and obtain cyanamide according to the following reaction.  $2 \text{CON}_2\text{H}_4 + \text{Na}_2 = 2 \text{CN}_2\text{H}_4 + 2 \text{NaOH} + \text{H}_2$ . Ammonium carbamate and ammonium carbonate behave similarly under like circumstances.

**On the action of Haloid Acids upon Hydrocyanic Acid.**  
(Vol. XL. p. 264). By L. CLAISEN, Ph. D. AND F. E. MATTHEWS, F.C.S.

After giving details of their experiments, the authors, conclude that, "when hydrocyanic acid unites with haloid acids in presence of ethers, a simple addition compound is not formed, but a more complex one, 2 mols. of hydrocyanic acid uniting with the haloid acid." The body  $2 \text{HCN} + 3 \text{HCl}$ . when acted upon by alcohol gave *formamidine*.

**On the action of Acetyl Chloride on Fumaric Acid.**  
(Vol. XL. p. 268). By W. H. PERKIN, F.R.S.

This paper gives some results of experiments in answer to the criticisms of Anschütz (Ber. 14, 2792) and C. Bennert (Ber. 15, 640). The author concludes that acetyl chloride acts upon fumaric acid in the same way as it does on other dibasic acids, forming the anhydride by the removal of a molecule of water, the anhydride in this case being maleic anhydride ; and as a secondary result, part of the maleic anhydride thus formed unites with the hydrochloric acid present and forms chlorosuccinic anhydride.

**On the Action of Acetone on Phenanthraquinone, both alone and in Presence of Ammonia.** (Vol. XL., p. 270.) By FRANCIS R. JAPP, M.A., Ph. D., and FRED. W. STREATFIELD.

Fifty grms. of phenanthraquinone were mixed with 60 grms. of acetone, and 40 cc. of strong aqueous ammonia. The quinone is converted into a white powder, and the temperature rises. When cool, ether was added, and the white powder was washed with ether.

The crude product was dissolved in boiling acetone (b. p. 56-58) containing a little aqueous ammonia. The filtered solution gave colorless rhomboidal laminæ. The fusing point could not be determined accurately; the body changes color at about 120° and fuses about 130°. Analysis leads to the formula  $C_{17}H_{11}NO_2$ . It dissolves readily in cold concentrated hydrochloric acid, the solution deposits a blue substance which was filtered out, and the filtrate treated with water. Water caused the deposition of fine white needles, which crystallized from ether gave large thin colorless blades, fusing at 89.5-9°, and containing no nitrogen or chlorine. Analysis gave formula  $C_{17}H_{14}O_2$ , according to the reaction  $C_{17}H_{11}NO_2 + OH_2 = C_{17}H_{14}O_2 + NH_3$ , since the hydrochloric acid filtrate from needles contains ammoniac chloride. The body  $C_{17}H_{14}O_2$  is insoluble in water; ether, alcohol and acetone dissolve it freely. In the alcoholic solution boiling converts it into phenanthraquinone; below 40° it can be crystallized from alcohol. By passing  $NH_3$  into the ether solution of the body  $C_{17}H_{14}O_2$ , the former body  $C_{17}H_{11}NO_2$  is obtained according to the reaction  $C_{17}H_{14}O_2 + NH_3 = C_{17}H_{11}NO_2 + OH_2$ . By heating the body  $C_{17}H_{14}O_2$ , it is decomposed into acetone and phenanthraquinone. This same body can be formed synthetically by heating phenanthraquinone with acetone in sealed tubes for four hours at 200°.

The authors give a number of theoretical considerations as to the constitution of the body formed, but seem to arrive at no definite conclusions. For the body  $C_{17}H_{14}O_2$  they suggest the name *acetonquin of phenanthrene*, and for  $C_{17}H_{11}NO_2$  the name *acetonquinimide of phenanthrene*. They will continue their studies on these bodies.

**A Study of Some of the Earth-metals contained in Samarskite.** (Vol. XL., p. 277.) By HENRY E. ROSCOE, V.P.R.S.

Author gives the details of experiments upon the separation of terbia, yttria, and the so-called phillippia in the form of formates by fractional precipitation. A very careful series of measurements of the so-called phillippian formate crystals as rhombic prisms is given, for comparison. A solution of the earths terbia, phillippia and yttria was passed through the processes of fractionation, and no oxide having an unalterable atomic weight of 122 could be detected. Author gives details of experiments which serve to prove that the formates of terbium and yttrium crystallize together in a form ascribed to phillippium.

**The Spectrum of Terbium.** (Vol. XL., p. 283.) By H. E. ROSCOE and A. SCHUSTER.

The spectrum was obtained with "a grating of Mr. Rutherford containing 17,290 lines to the inch, and all measurements were taken in the spectrum of the second order. The focal length of the observing telescope was 17.4 inches, and the measurements were taken by means of a micrometer eye-piece, the screw of which had 100 turns to the inch." A table of the lines observed is given, showing their wave-lengths and relative intensity.

**On the Behavior of Zinc, Magnesium, and Iron as Reducing Agents with Acidulated Solutions of Ferric Salts.** (Vol. XL., p. 287). By T. E. THORPE, F.R.S.

In the case of zinc the author states: "It results from these observations that, provided there is sufficient acid present to dissolve the zinc, the maximum reducing action possible with a given weight of metal is obtained by concentrating the ferric sulphate solution and diminishing the amount of free acid. The effect of concentration is to multiply the chances of contact of the hydrogen and ferric sulphate molecules; increase of temperature brings about the same result, by augmenting the molecular movement within the liquid." By using platinum in contact with the zinc, the time of solution is diminished, but very little difference in the reducing effect is observed.

With magnesium the amount of reduction is only  $\frac{1}{4}$  to  $\frac{1}{3}$  of that effected by zinc under corresponding conditions. The presence of platinum but slightly augments the reducing power of magnesium.

With iron the rate of solution is extremely slow, and the reducing action *apparently* decreases with the temperature. In contact with platinum both the rate of solution and the reducing action are increased; the action is *decreased*, however, on warming the liquid.

The author remarks: "These experiments seem to me to be in entire harmony with the view that the reducing action of so-called nascent hydrogen is connected with the existence of this body in the atomic state, and that any conditions tending to prolong the duration of this atomic condition augment the reducing power of the hydrogen."

**Note on the Action of the Oxychlorides of Sulphur on Silver Nitrate.** (Vol. XL., p. 297). By T. E. THORPE, F.R.S.

Thionyl chloride  $\text{SOCl}_2$  with silver nitrate gives nitrosulphonic chloride and silver chloride. Sulphuryl chloride  $\text{SO}_2\text{Cl}_2$  has no

action. Chlorosulphonic acid  $\text{SO}_2\text{OHCl}$  acts violently on silver nitrate, forming silver chloride and nitrosulphonic acid (chamber crystals.)

**On the Action of Thiophosphoryl Chloride upon Silver Nitrate.** (Vol. XL., p. 297). By T. E. THORPE, Ph. D., F. R. S., and SEPTIMUS DYSON, Esq.

By adding thiophosphoryl chloride drop by drop to dry silver nitrate and condensing the products of the action, the authors obtained nitrogen peroxide, nitric oxide and nitroxyl chloride, and *nitrosulphonic anhydride*. The authors say: "We have no hope, therefore, of being able to form a mixed anhydride, on the type of nitric anhydride in which more or less of the oxygen is replaced by sulphur, by the substitution of thiophosphoryl chloride in Odet and Vignon's reaction."

**Experiments on the action of Potassium Amalgam Sulphuretted Hydrogen, and Potassic Hydrate respectively, on Tetra and Pentathionate of Potassium.** (Vol. XL., p. 300.) By VIVIAN LEWES, Royal Naval College.

By the action of potassium amalgam upon potassium tetrathionate in solution the thiosulphate ( $\text{K}_2\text{S}_2\text{O}_3$ ) is found when no excess of amalgam is used; in the latter case sulphide is formed. With potassic pentathionate potassium amalgam gives first tetrathionate and sulphur, then thiosulphate.

By the action of sulphuretted hydrogen upon potassic tetrathionate, the thiosulphate is formed and sulphur separates.

By the action of potassic hydrate upon a solution of potassic pentathionate thiosulphate, sulphite and sulphur result from the decomposition. In the case of potassic hydrate and tetrathionate the results are thiosulphate and sulphite.

**On the Estimation of Retrograde Phosphates.** (Vol. XL., p. 306.) By FRED. J. LLOYD.

Author reviews the work of Fresenius, Neubauer, Luck and others in this field. He then proceeds to find out the relative dissolving powers of citric acid, partly neutralized, wholly neutralized, and more than neutralized by ammonia, upon the phosphates and other substances present in manures. He concludes that no satisfactory determination of retrograde phosphates can be made by an acid or slightly acid solution of ammonium citrate, owing to the powerful action upon other substances. From another set of experiments the author states that an ammonical solution of ammonium citrate, no matter what its

strength, whilst dissolving precipitated calcic phosphate, does not act upon mineral phosphates, bone ash, and but slightly upon raw bones. This paper is a most valuable one, as the author gives a number of details of experiments upon the methods of other chemists, and a comparison with that proposed by himself.

**Action of Heat upon Mercuric Chloride under low pressure.** (Vol. XL., p. 322). By THOS. CARNELLEY, D.Sc.

The general results of the author's experiments are: "When mercuric chloride is strongly heated under pressures less than the critical pressure, then the temperatures attained by the salt as indicated by the thermometer, the turpentine calorimeter, and the petroleum or benzine calorimeter, are as follows:

*By the thermometer.*—As a compact powder the salt rises  $21^{\circ}$  C., and sometimes  $50^{\circ}$  C., above the melting point. When the salt is solidified around the thermometer bulb it rises  $15^{\circ}$  C. above the melting point. *By the turpentine calorimeter.*—the temperature rises  $100^{\circ}$  C. above the melting point. *By the petroleum calorimeter.*—The temperature cannot be obtained above the melting point; but is generally a few degrees less.

The author concludes that the high temperature registered by the turpentine calorimeter is due to the heat developed by the action of the mercuric chloride upon the hydrocarbon.

**On the Constitution of Amarine and Lophine.** Vol. XL., p. 323. By FRANCIS K. JAPP, M. A, Ph.D. and H. H. ROBINSON.

From a consideration of Emil Fischer and H. Troshke (Ber. 13. 706) and a comparison with the formula obtained by one of the authors and Mr. Streatland (Jour. Chem. Soc., xl. p. 149) for the compounds formed by the action of hydroxyaldehydes and ammonia upon phenanthraquinone, an unmistakable resemblance was noticed. Acting upon this resemblance, and submitting benzil to the action of parahydroxybenzaldehyde in presence of ammonia, they obtained the body  $C_{11}H_{10}N_2O$ , which by distillation with zinc dust in a current of hydrogen gave *lophine*. The body  $C_{11}H_{10}N_2O$  is *parahydroxylophine*, and lophine is considered to be one of Hubner's anhydro-bases, and is called *anhydrobenzayldiamidostilbene*. From these facts and others connected with amarine, which differs from lophine in that it contains two more atoms of hydrogen, the authors give new constitutional formulæ for both these bodies.

Abstracts from *Berichte der Deutschen Chemischen Gesellschaft*, by Percy Neymann. Ph. B

**On a New Combination of Carbon with Sulphur and Bromine.** CARL HELL and FR. UHECH, (Vol. XV, p. 283.)

A mixture of bisulphide of carbon and bromine (the proportion  $1\text{CS}_2 : 2 \text{Br}_2$  has been found best) after having been left to itself several days and then subjected to a slow distillation leaves a less volatile brownish-red oily residue which does not solidify until brought in contact with water, but then gradually crystallizes. A more rapid method is to cover the oily residue with alcohol instead of with water, or to dissolve the residue in ether and add alcohol. The composition of the new body, termed by the authors carbotrithiohexabromide corresponds to the formula  $\text{C}_2\text{S}_3\text{Br}_6$ .

The action of concentrated caustic alkali at waterbath temperature and presence of a current of hydrogen to prevent oxidation corresponds exactly to the formula :

$\text{C}_2\text{S}_3\text{Br}_6 + 12\text{NaOH} = 2\text{Na}_2\text{CO}_3 + \text{Na}_2\text{S}_3 + 6\text{NaBr} + 6\text{H}_2\text{O}$ . The constitution of the new combination cannot be fixed upon either from the conditions under which it is formed or from the products of decomposition.

**On the action of Amines upon Chinones.** H. FINCKE. (Vol. XV. p. 279), continued from Vol. XIV. p. 1310, derivatives.

**On Tropin.** G. MERLING. (Vol. XV. p. 287), continued from Vol. XIV. p. 1829.

The author starting with 40 grms. tropin obtained only 1 gram. of the neutral oil by the distillation of the dimethyltropinhydroxide. Oxidation of tropin with permanganate of potash in alkaline solution gives rise to a base which by the separation of one atom of carbon as carbonic acid and the formation of a nitroso compound by the action of nitrous acid and also by the regeneration tropin when treated with methyl iodide, must be characterised as a secondary base. Excess of oxidizing agent must be avoided.

Oxidation of tropin by means of bichromate of potash and sulphuric acid or by chromic acid produces (without the separation of carbonic acid) a well characterised dibasic acid of the formula  $\text{C}_8\text{H}_{11}$ ,

COOH.

$\text{NO}_2 = (\text{C}_8\text{H}_{11}\text{N})$ .

COOH.

The author expects at some future day, to be able to contribute something more certain on the constitution of tropin.

**On Terebenthenic acid.** W. ROSE. (Vol. XV. p. 293).

Sodium reacts with terebenthenic acid ether  $C_9H_{14}O_4$  and generates hydrogen; the white crystals which are the product of the reaction are not a sodium combination, analogous to that obtained by J. Bredt\* from isocapralacton, but it is the sodium salt of the acid teraconicacidether after liberating the ether by means of acids this yields teraconic acid ( $C_7H_{10}O_4$ ) by saponification which Geissler found among the products of distillation of terebenthenic acid. Terebenthenic acid ether treated in alcoholic solution with one equivalent of sodium ethylate also produces the sodium salt of the acid teraconic acid ether, a second equivalent of ethylate of sodium acts as a saponifying agent upon the primary salt and teraconate of sodium is produced. It was also shown that unless a certain quantity of water was present succinate of sodium was produced. In order to explain the formation of teraconic acid from terebenthenic acid it must be accepted that by the action of sodium upon terebenthenic acidether a sodium compound is at first produced, analogous to the sodium acetosuccinicacidether but that this is immediately converted into the sodium salt of the acid teraconicacidether.

**Lecture Experiments.** V. MEYER. (Vol. XV. p. 297).

Demonstrations: Pure water is not colorless but blue. Transformation of yellow phosphorous into the red by heating in vapor of diphenylamin. Description of apparatus.

**On a new Isomer of Orcin.** EDM. KNECHT. (Vol. XV. p. 298).

Preparation of bioxytoluol (cresorcin) by consecutive steps from dinitrotoluol, nitrocresol, amidocresol, bioxytoluol. The new body which is a genuine homologue of resorcin (because it also has the OH groups in the meta position) is easily mistaken for resorcin. It differs from resorcin in its crystallographic forms. The author proposes the name cresorcin in order to point out its derivation from cresol and at the same time its great similarity to resorcin.

**On Phosphorouspentasulphide.** H. GOLDSCHMIDT. (Vol. XV., p. 303.) Determination of boiling point by means of Meyer's and Goldschmidt's air thermometer. Mean boiling point for barometric pressure 728.5-734 mm., was found to be  $518^{\circ}C$ .

**On Naphtylsulphuric Acid.** R. WIETZKI. (Vol. XV., p. 305.)

It has of late repeatedly been observed that the naphthols in

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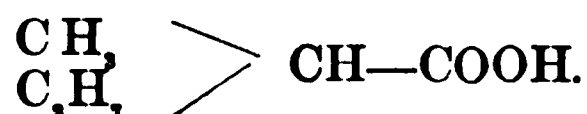
\*J. Bredt. Ann. Chem. 208.58.



reference to the ease with which their O H groups can be replaced, are more similar to the alcohols of the fatty series than to the phenols. Pulverized and dried naphthol rubbed in a mortar with  $1\frac{1}{2}$  to 2 parts of common conc. sulphuric acid at first forms a thick syrupy mass, no perceptible rise of temperature being observed. This takes place, however, after a time, and the mass crystallizes. The mass dissolved in water, neutralized with soda and a larger quantity of salt added, separates fine colorless crystals. From the reactions and characteristics of this body it is shown that it is analogous to the class of bodies known as ethersulphuric acids. The author states that an ethersulphuric acid of alpha-naphthol also exists, but he has not been able to isolate its salts. These acids cannot be combined as such with diazo compounds.

**On the Capronic Acid occurring in Resin Oil.** WERNER KELBE and CONSTANTIN WARTH. (Vol. XV., p. 308.)

Capronic acid was obtained from the mixture of acids occurring in resin oil by fractional distillation of the ethers of these acids. The fractions were taken at  $150^{\circ}$ - $155^{\circ}$ C. After saponifying the ether with caustic potash, the capronic acid was liberated by means of hydrochloric acid, and the free capronic acid was driven off with steam. The calcium salt was used for purification. The author finds from the properties and reactions of this capronic acid that it is identical with methylpropylacetic acid :



**On the Action of Pentachloride of Phosphorus on Alpha-Naphtholsulphonic Acid.** AD. CLAUS and H. OEHLER. (Vol. XV., p. 312.)

The action of pentachloride of phosphorus upon the sulphonic acid of alpha-naphthol differs from that upon beta-naphtholsulphonic acid in as much as the formation does not take place by ether-like combinations between the alpha-naphtholhydroxyl and sulphonic groups or from phosphoric acid ethers. Below  $100^{\circ}$  alpha-naphtholsulphonic chloride is formed. If the mixture of the alpha-naphtholsulphonic salt and pentachloride of phosphorus is heated to  $100^{\circ}$  or  $150^{\circ}$ , two new bodies—chlornaphthol and dichlornaphthaline can be isolated.

**Synthesis of the Homologues of Anilin from Bromanilines, Bromtoluidines, etc., by the action of Sodium and**



**Alkylbromides in Ethereal Solution.** AD. CLAUS. (Vol. XV., p. 315).

Xylidine from Bromtoluidine. Bromparatoluidine was employed. The effect of sodium on a mixture of this body with an excess of methyliodide in ethereal solution takes place without the formation of much pitch like substance. The oil obtained by the usual method of acidifying with hydrochloric acid, and liberating the base with caustic potash, was found not to be pure xylidine. Azotoluol and toluidin are formed in small quantities. The base or bases obtained by the same process from normal propylbromide and parabromaniline remain to be experimented upon.

**On Dichlornaphtralin and Chlornaphthol from Beta-Naphtholsulphonic Acid.** AD. CLAUS and M. DEHNE. (Vol. XV., p. 319).

The authors conclude as to the difference between alpha and beta naphtholsulphonic acid as follows: the tendency of the former to form basic salts is scarcely at all shared by the latter; while from the first by means of nitric acid, dinitronaphthol can easily be formed, the latter does *not* yield sulphuric acid when boiled with nitric acid, but apparently produces a new *nitrated* sulphonic acid. By treating with chlorate of potash and hydrochloric acid the alpha-naphtholsulphonic acid is converted into dichlornaphthachinon in the cold, while the same reagent acts upon beta-naphthosulphonic acid only at boiling heat, and then but very slowly.

**On the Quantity of Furfurol contained in Fermented Liquids.** K. FOERSTER. (Vol. XV., p. 322).

Small quantities were found in the distillates of beer and pure wines. It was also found in the liquid filtered, but not distilled from a fermented solution of 300 grains of starch-sugar (artificial dextrose). The author sums up as follows:

1. That all conclusions on the presence of amylic alcohol by the aid of Jorissen's reaction are wrong.
2. That no means have yet been found by which small quantities of amylic alcohol can be quantitatively determined with safety in fermented solutions.
3. That all suspicions referring to the use of starch sugar for improving the wine because the quantity of fusel oil was found larger, are either uncertain or false.

**Benzol, Dipropargyl and Acetylene.** Constitution of Benzol. JUL. THOMSEN. (Vol. XV., p. 328).

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Information .....	SECRET
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Information .....	SECRET
1. Summary of Situation .....	SECRET
2. Summary of Index .....	SECRET
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Information .....	SECRET

But we were not able to determine the direction the  
 car was going of which we suspected was east. The  
 vehicle was in the southwest corner of the intersection  
 and was going south.

**Das Landwirthsch. P. FACHLEHRE mit H. SCHNEIDER** VII  
28. J. 642, Ausgabe vom Vol. XIV. u. 1914

On the Action Produced from Sulpho-Carbonated by Addition of Iodine Alkalies. W. W. Vol. IV, p. 325.

Investigation of the materials of construction of vessels of various types  
and the investigation of the materials of construction of vessels of various types

**On Anthracylamin. H. Kerner Vol. IV, p. 231.**  
From anthracylamin, by oxidation, two new substances

Interest in using sodium to enhance the limit of saturation of polymers with styrene monomer, "Orange No. 3" of Polymer Associates, Inc., and "Heliocaine" have been employed. The apparent addition of these substances is yellow, but it turns red when in contact with free acids only, as demonstrated by W. MEIER for "Tropoline" (Platinum Phenylamidoazobenzene).

**Abstracts from the Complex Rendus, By A. BOUQUARD.**

**Magnesium Oxychlorides.** BY G. ANDER. No. 7, Feb. 13th, 1882).

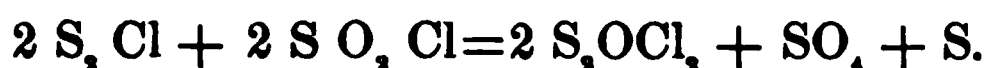
Prepared by heating 500 grammes of water, 400 gms. of crystallized magnesium chloride, and adding to the mass when boiling 20 gm. calcined magnesia. A clear liquid is separated by filtration,

and after some time a deposit having the appearance of fine needles is formed, having the composition  $\text{Mg. Cl}_2$ ,  $\text{Mg. O}$ ,  $16 \text{ H}_2 \text{ O}$ . Dried in vacuo until a constant weight is obtained, it gives  $\text{Mg. Cl}_2$ ,  $\text{MgO}$ .  $6 \text{ H}_2 \text{ O}$ .

These two oxychlorides are immediately decomposed by water or alcohol.

**A New Oxychloride of Sulphur.** BY J. OGIER. (No. 7, Feb. 13th, 1882).

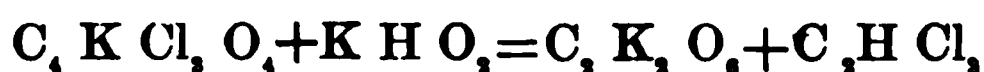
This new oxychloride has been prepared by treating in strong sealed tubes a mixture of equal weights of sulphur chloride and sulfuryl chloride. Boiling point  $60^\circ$ — $61^\circ \text{ C}$ . Very easily decomposed by heat. Composition  $\text{S}_2 \text{ O Cl}_2$ . Its formation is represented by the equation



Vapor density with Meyer apparatus at  $100^\circ \text{ C}$ . 3.28 ; 3.84 ; 3.75. Dumas' method at  $60^\circ \text{ C}$ ., under a pressure of 10 cc. of mercury gives 3.9.

**Action of Potassium Cyanide upon Potassium Trichloracetate.** BY E. BOURGOIN. (No. 7, Feb. 13th, 1882).

The action of potassium cyanide upon a concentrated solution of potassium trichloracetate is similar to the action of an alkali upon the trichloracetate.



**Heat Developed During the Formation of Ferricyanhydric Acid.** BY M. JOANNIS. (No. 7, Feb 13th, 1882).

(The articles upon Thermochemistry do not admit of abstraction).

**On Galactin.** BY A. MUNTZ. (No. 7, Feb. 13th, 1882).  
Extracted from the seeds of Lucerne (*Medicago arborea* L.)

White, translucent mass, swelling and slowly dissolved by water, giving a limpid solution ; is not precipitated by neutral lead acetate, but by basic acetate. Same composition as the gums. Dextrogyrate. Treated with nitric acid, gives mucic acid.

**Aconitates.** BY E. GUINOCHET. (No. 7, Feb. 13th, 1882).

(This article does admit of abstraction.)

**Double Salts of Mercury.** (Thermochemistry.) BY M. BERTHELOT. (No. 8, Feb. 20th, 1882).

**On Ferrocyanhydric Acid.** (Thermochemistry.) BY M. JOANNIS. (No. 8, Feb. 20th, 1882).

**Upon the Saturation of Phosphoric Acid by Bases and upon Chemical Neutrality.** By A. JOLY. (No. 8, Feb. 20th, 1882.)

**Ferri cyanhydric Acid.** By M. JOANNIS. (No. 8 Feb. 20, 1882). (Thermochemistry.)

**Action of Iodine upon Naphthaline at a High Temperature.** By A. BLEUNARD and G. VRAU. (No. 8 Feb. 20, 1882).

Naphthaline is dehydrogenized by iodine; hydriodic acid is formed, and a new compound  $(C_{10}H_8)_x$  has been obtained, which represents naphthaline less two atoms of hydrogen.

**Double Salts of Mercury.** By M. BERTHELOT. (No. 9 Feb. 27, 1882). (Thermochemistry.)

**Analysis of a Volcanic Ash from Mt. Etna.** By L. RICCIARDI. (No. 9 Feb. 27, 1882).

Silica.....	37.82
Sulphuric acid.....	20.57
Alumina .....	9.97
Protonide of iron.....	14.05
Lime.....	11.98
Magnesia.....	3.64
Chlorine .....	1.02
Soda and Potassa.....	0.95
	<hr/>
	100.00

**Normal Carbonic Acid of the Atmosphere.** By M. DUMAS. (No. 10 March 6, 1882.)

Among the causes which concur to equilibrate the quantity of carbonic acid in the atmosphere, the author relates an explanation given lately by Schlosing, and derived from the principle of dissociation. The proportion of carbonic acid contained in the atmosphere is in proportion with the quantity of calcium bi-carbonate dissolved in the waters of the seas. When the quantity of carbonic acid decreases, the marine calcium bi-carbonate is dissociated; half of its carbonic acid gas goes in the atmosphere and neutral calcium carbonate is deposited. When the aqueous vapor is condensed in the atmosphere, it now dissolves a portion of the carbonic acid present, and when the rain, so charged with carbonic acid, comes in contact with the earth it dissolves lime, calcium bi-carbonate is formed and carried to the sea.

The variation of the proportion of carbonic acid in the atmosphere is very small; this variation is about  $\frac{3}{10000}$  in volume.

**Upon the Double Decompositions of the Haloid Salts of Mercury by the Hydracids and the Haloids Salts of Potassium.** By M. BERTHELOT. (No. 10 March 6, 1882). (Thermochemistry.)

**The formation of two Dibasic Acids, Sebacic and Suberic During the Distillation of Crude Fatty Acid in a Current of Superheated Steam.** By A. CAHOUS AND E. DEMARCY. (No. 10 March 6, 1882).

The distillation of crude fatty acids in a current of superheated steam transforms these acids into saturated hydrocarbons, homologous with marsh gas, in acids of the acetic group belonging to the normal series, and finally into two dibasic acids, suberic and sebacic acids, homologous with succinic acid.

**Double Decompositions of the Haloid Salts of Mercury.** By M. BERTHELOT. (No. 11 March 13, 1882). (Thermochemistry).

**Crystallized Oxychloride of Gallium.** By L. DE BOISBAUDRAN. (No. 11 Mar. 13th, 1882). After three years a sample of gelatinous hydrated chloride of gallium was transformed in small crystals surrounded by a liquid. The analysis of the crystals (octahedrons) has been given.  $(\text{Ga}_2\text{Cl}_6 + 12 \text{H}_2\text{O}) + 2 (\text{Ga}_2\text{O}_3 + \text{H}_2\text{O})$ .

**Heat of Formation of Ferricyanhydric Acid, and of some Ferrocyanides.** By M. JOANNIS. (No. 11. Mar. 13th, 1882). (Thermo-chemistry).

**Products of Distillation of Colophony.** By A. RENARD. (No. 11. Mar. 13th, 1882). The distillate of colophony boiling at about  $150^\circ \text{C}$ . contains three hydrocarbons, a terebenthen  $\text{C}_{10}\text{H}_{16}$  and two isomeric hydrocarbons  $\text{C}_{10}\text{H}_{16}$ . These three hydrocarbons can not be separated by distillation; their mixture is laevogyrate.

**Dichloride of Camphor.** By P. CAZENEUVE. (No. 11. Mar. 13th 1882.) Obtained by the action of dry chlorine upon camphor, dissolved in absolute alcohol. Corresponds to  $\text{C}_{15}\text{H}_{22}\text{Cl}_2\text{O}$ .

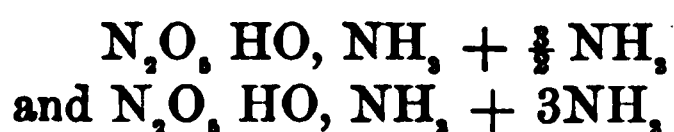
**Essential Oil of Licari Kanali.** By H. MORIN. (No. 11. Mar. 13th, 1882).

**Determination of Tannic and Oenogallic Acid in Wine.** By F. JEAN. (No. 11. Mar. 13th, 1882).

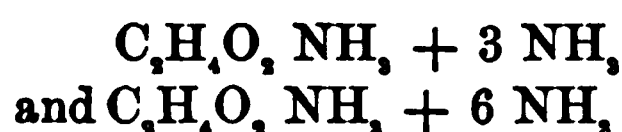
**Gastric Digestion.** By E. DUCLAUX. (No. 11. Mar. 13th, 1882).

**Double Decompositions of the Haloids Salts of Mercury.** Conclusions. By M. BERTHELOT. (No. 12. Mar. 20th, 1882) (Thermo-chemistry).

**New Combinations of Nitric Acid and Acetic Acid with Ammonia.** By L. TROOST. (No. 12. Mar. 20, 1882). By the action of dry ammonia upon dry nitrate of ammonium two nitrates were obtained.



The new combinations of acetic acid and ammonia are



**Action of Acid Solutions upon Protoxide of Iron.** By A. DITTE. (No. 12. Mar. 20th, 1882).

**Action of Ozone upon the Salts Manganese.** By M. MAQUENNE. (No. 12. Mar. 20th, 1882). Ozone produces in the solutions of salts of protoxide of manganese a brown precipitate of hydrated oxide of manganese. In acidifying the solutions, other products, such as permanganic acid, are formed.

**Heat of Formation Sulphocyanic Acid and of some Sulphocantes.** By M. JOANNIS. (No. 12. Mar. 20th, 1882). (Thermo-Chemistry).

**Clarification of Champagne Wines.** By F. JEAN. (No. 12. Mar. 20th, 1882). Tannic acid in solution in the must is titrated with a solution of iodine. When the amount of tannic acid is known an equivalent quantity of gelatine is added, the gelatine and tannic acid form with albuminoid matter an insoluble precipitate—82.8 of tannic acid requires 100 of gelatine.

**Kola Nut, Seeds of Sterculia Acuminata.** By E. HECKEL and F. SCHLAGDENHAUFFER. (No. 12. March 20, 1882.)

Caffein.....	2.348	} Soluble in Chloroform.
Theobromin.....	0.023	
Tannic acid.....	0.027	
Fat.....	0.585	

Tannic acid.....	1.591	} Soluble in Alcohol.
Coloring matter, (red).....	1.290	
Glucose.....	2,875	
Salts.....	0.070	
Starch.....	33.754	
Gum.....	3.040	
Coloring matter.....	2.561	
Proteids.....	6.761	
Ashes.....	3.325	
Water.....	11.919	
Cellulose.....	29.831	
Total.....	100,000	

**Double Decompositions of Haloid Salts of Silver.** By M. BERTHELOT. (No. 13, March 27th, 1882), (Thermochemistry).

**Researches Upon Ozone.** By M. MAILFERT. (No. 13. March 27th, 1882). The object of these researches is the action of Ozone upon organic matter, and upon several oxides and metallic sulphides, and also upon salts from which the bases can be suboxodized.

**Action of Alkaline Solutions Upon Protoxide of Tin.** By A. DITTE. (No. 13. March 27th, 1882.)

**Upon Campholurethane.** By A. HALLER. (No. 13. March 27th, 1882.)

**Preparation of Pure Carbon for Electric Light.** By M. JACQUELAIN. (No. 12. March 27th, 1882.) The impurities in carbon are removed by treatment with chlorine, caustic soda or hydrofluoric acid, and the carbon is then washed in water.

**Haloids Salts of Silver and Potassium.** By M. BERTHELOT. (No. 14. April 3d, 1882).

**Union of Free Hydrogen with Ethylen.** By M. BERTHELOT. (No. 14. April 3d, 1882).

The combination is limited by the dissociation of the hydrides, and the degree of dissociation varies rapidly with the temperature.

**Effects of Compression upon the Hardness of Steel.** By M. LAN. (No. 14. April 3d, 1882).

Steel is hardened by compression. In compressed steel the pro-

portion of combined carbon is increased and the proportion of free carbon diminished, as shown by the following determinations:

	Compressed.		Non-Compressed.
Carbon, total,	0.70 per ct.		0.70
Combined carbon, A,	0.60 "	} Average 0.585	0.49
" " B,	0.59 "		0.50
" " C,	0.55 "		0.47
" " D,	0.60 "		0.50
Free carbon (by difference)	0.115		0.21

**Upon the Composition of Hydrated Carbonic Acid.** By M. WROBLENSKI. (No. 14. April 3d, 1882).

At 0°C and under the pressure of about 16atm. hydrated carbonic acid has the composition  $\text{CO}_2\cdot 8\text{H}_2\text{O}$ .

**Synthesis of Quinine.** By E. G. MAUMENE. (No. 14. April 3d, 1882).

The author states that having discovered the compound  $\text{NH}_2$ , he has been able to prepare artificially some very pure sulphate of quinine, but that before describing his process he will make some comparative experiments in order to test the medical properties of this artificially prepared quinine.

**Action of Fuming Nitric Acid Hydrochloric Acid, upon Pilocarpin.** By P. CHASTAING. (No. 14. April 3d, 1882).

Fuming nitric acid transforms pilocarpin into nitrate of Jaborandin  $\text{C}_{10}\text{H}_{11}\text{N}_2\text{O}_4\text{NHO}_2$ .

By the action of hydrochloric acid pilocarpin is transformed into chloride of jaborandin.

**Transformation of Carbon Oxysulphide into Ordinary and Sulphuretted Ureas.** By M. BERTHELOT. (No. 16. April 17th, 1882).

**Chemical Monography of the Cucurbitaces of Uruguay.** By F. SACC. No. 16. (April 16th, 1882).

The author gives several analyses of the melons coming to the Montevideo market, also the analysis of the roots of Briony, which are often mistaken for wild potatoes. They have the following composition:

Pectic acid.....	0.33 per cent.
Lignine .....	9.65 "
Inulin .....,....	5.32 "
Grape sugar.....	4.86 "
Water and loss .....	79.84 "
	<hr/>
	100.00



**Separation of Gallium.** By M. LECOQ DE BOISBAUDRAN. (No. 17. April 24th, 1882).

**Upon the Atomic Weight of Carbon by the Combustion of Diamond.** By H. E. ROSCOE. (No. 17. April 24th, 1882).

African diamonds were employed. They did not yield a trace of hydrogen. Average of six experiments 11.07, oxygen being 15.96.

**Decomposition of Lead Salts by Alkalies.** By A. DITTE. (No. 17. April 24th, 1882).

**Researches upon Ozone.** By M. MAILFERT. (No. 17. April 24th, 1882.)

A study of the action of ozone upon sulphur, selenium, tellurium, sulphides and some organic matters.

**Insoluble Modification of Pepsine.** By A. GAUTIER. No. 17. April 24th, 1882).

**Oxydation of Pyrogallol in the Presence of Gum Arabic.** By P. de CLERMONT AND P. CHAUTARD. No. 18. (May 1st, 1882). The produce of oxidation is Purpurogallin, having the appearance of yellow needles  $C_{10}H_{10}O_6$ .

**Chemical Studies of Several Products of Uruguay.** By F. SACC. No. 18. (May 1st, 1882).

**Composition and Atomic Weight of Pernitric Acid.** By P. HAUTEFEUILLE AND G. CHAPIUS. No. 19. (May 8th, 1882).

**Action of Potassa Upon Lead Oxide.** By A. DITTE. No. 19. (May 8th, 1882).

**New Compounds of Carbon and Silicium.** By A. COLSON. No. 19. (May 8th, 1882).

This new compound is made by means of a stream of hydrogen saturated with benzol, passing over silicium placed in a red-hot porcelain tube.

**Upon the Homologous and Isomeric Rosanilin.** By A. ROSENSTIEHL AND M. GERBER. No. 19. (May 8th, 1882).

**Composition of Ashes from Vesuvius.** By L. RICCIARDI. No. 19. (May 8th, 1882).

Eruption of February 25th, 1882.

Silica .....	47.84
Anhydr. Sulphuric Acid.....	0.17
“ Phosphoric Acid.....	1.83

Chlorine .....	1.32
Alumina.....	18.67
Protoxide of Iron.....	5.07
Peroxide of " .....	4.38
Lime .....	9.42
Magnesia.....	3.77
Potassa.....	5.64
Soda.....	2.04
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	100.15

**Studies of the Antiseptic Properties of Salicylic Acid.**  
 BY E. ROBINET AND H. PELLET. No. 19. (May 8th, 1882).

**Upon Some Reactions of Mercuric Chloride.** BY H. DEBRAY. No. 18. (May 1st, 1882).

Mercuric chloride is reduced to mercurous chloride by the solution of sulphurous acid, the presence of sodium chloride prevents the reduction.

When a solution of an alkali is mixed slowly with a solution of mercuric chloride, the precipitate varies in color from yellow to black, especially in warm liquids, these different colorations are produced by the formation of oxychlorides of variable composition. With an excess of alkali, all the oxychlorides are destroyed, and the precipitate is formed only of yellow oxide of mercury. The presence of sodium chloride prevents the formation of these intermediary products.

**Liquefied Gases, and Particularly Ethylen, for the Production of Low Temperatures.** BY L. CAILLETET. No. 18. (May 1st, 1882).

The evaporation of ethylen in an open vessel produces a temperature of  $-105^{\circ}$  C. ; this temperature is far lower than that produced by nitrous oxide which boils at  $-88^{\circ}$  C.

**Separation of Gallium.** BY M. LECOQ de BOISBAUDRAN. No. 18. (May 1st, 1882).

**Liquefaction of Ozone.** BY P. HAUTEFEUILLE AND G. CHAPUIS. No. 18. (May 1st, 1882).

Drops of liquefied ozone had been obtained with a Cailletet's apparatus, the liquid is dark-blue indigo, and had been preserved for thirty minutes under a pressure of 75 atm. Its vaporization is

not very rapid even under atmospheric pressure. The liquefaction has been obtained by compressing a mixture of oxygen and ozone under 125 atm., the refrigerating liquid being ethylen, and the temperature was below—100 C°.

**Action of Insoluble Metallic Sulphides upon a Solution of Acid Sulphate of Nickel in the presence of H<sub>2</sub>S.** BY H. BAUBIGNY. No. 18. (May 1st, 1882).

**Synthesis of Organic Compounds by Means of Electrolysis of Water, Acid, Alkaline and Alcoholic Solution with Carbon Electrodes.** BY A. BARTOLI and G. PAPASOGLI. No. 20. (May 15, 1882).

**On the Absorption of Gases by Platinum.** BY M. BERTHELOT. No. 21. (May 22, 1882).

**Action of Peroxide of Hydrogen upon Organic Matter and Fermentation.** BY P. BERT and P. REGNARD. No. 21. (May 22, 1882). Very diluted peroxide of hydrogen stops the fermentations produced by living organisms and the putrefaction of all substances which do not decompose it. It has no action upon the diastasic fermentations. Diluted peroxide of hydrogen is not destroyed by fats, amylaceous matter, soluble ferments, egg albumen, casein, peptones, creatin, creatinin, or urea, but is quickly decomposed by musclin, fibrin and generally by vegetable nitrogenized matter.

**Separation of Gallium.** BY LECOQ DE BOISBAUDRAN. No. 22. (May 29, 1882). Analytical methods for the separation of gallium from glucinium, cerium, lanthanum, didymium, samarium, yttrium, erbium, holmium, thalium, iron and thorium.

**Determination of Glycerin in Fatty Matters.** BY J. DAVID. No. 22. (May 29, 1882). The saponification is made with crystals of barium hydrate, alcohol and water; alcohol is separated by boiling and barium with sulphuric acid. The clear liquid is evaporated to a determined volume, its sp. gr. taken and compared with the sp. gr. of a solution of glycerin and water of a known composition.

**Double Salts Prepared by Fusion.** BY MM. BERTHELOT and FLOSBEY. No. 23. (June 5, 1882).

**Oxychloride of Zinc.** BY G. ANDRE. No. 23. (June 5, 1882). A new oxychloride of zinc.  $2 \text{ZnCl} \cdot 5 \text{Zn}_2\text{O} \cdot 26 \text{H}_2\text{O}$  is described.

**Action of Carbon Di-Sulphide upon Silicium.** By A. COLSON. No. 23. (June 5, 1882).

**Preliminary Note on Didymium.** By T. CLEVE. No. 23. (June 5, 1882). The differences obtained in the determinations of the atomic weight of didymium have induced the author to believe in the existence of a new element which he calls beta-didymium.

**Explosible Alloys of Zinc with the Platinum Metals.** By SAINTE-CLAIRE DEVILLE and H. DEBRAY. No. 24. (June 12, 1882).

**Separation of Gallium.** By LECOQ DE BOISBAUDRAN. No. 25. (June 19, 1882). Separation of gallium from zirconium, manganese and zinc.

**Upon Silicium.** By P. SCHUTZENBERGER and A. COLSON. No. 26. (June 26, 1882).

**On the Compound  $\text{NH}_3$ .** By M. COMBES. No. 26. June 26, 1882). According to the author, the compound  $\text{NH}_3$  has not been obtained.  $\text{NH}_3$  and carbonic acid had been formed in the reaction alluded to by M. Maumene.

**On Didymium.** By B. BRAUNER. No. 26. (June 26, 1882). Ordinary didymium is a mixture of three elements—didymium, beta-didymium and perhaps samarium.

**Action of Peroxide of Hydrogen upon the Coloring Matter of Blood and upon Hematosin.** By A. BECHAMP. No. 26. (June 26, 1882). Description of the action of peroxide of hydrogen upon hemoglobin and hematosin and upon the white matter of hemoglobin.

**Electrolysis of Hydrogen Dioxide.** By M. BERTHELOT. (No. 1, July 3d, 1882.)

**Remarks on Didymium.** By P. T. CLEVES. (No. 1, July 3d, 1882.) In a preliminary note the author suggested the existence of a new element which he named Beta-Didymium, but further experiments have shown the non-existence of this new element.

**Action of Hydrogen Sulphide upon Nickel Chloride.** By H. BAUBIGNY. (No. 1, July 3d, 1882.) The transformation into sulphides of some metallic chlorides is more difficult than the transformation of the corresponding sulphates, under the same conditions,

by the action of hydrogen sulphide. Equivalent weights of sulphate and chloride of nickel being taken and submitted under the same conditions to the action of hydrogen sulphide, it has been observed that the conversion of the sulphate into sulphide was quicker than for the chloride.

**Isomery of Cuprous Sulphites.** By M. ETARD. (No. 1, July 3d, 1882.)

**Reduction of Some Silver Ores by Hydrogen and the Wet Way.** By P. LAUR. (No. 1, July 3d, 1882.) The silver ore, sulphide, chloride, iodide or bromide in fine powder is placed in a cast iron pan with a weak alkaline solution (1 part of soda for 100 of water) and an amalgam of 3 parts of tin for 100 of mercury and submitted to ebullition, nascent hydrogen decomposes the argentiferous compounds, the silver forms an amalgams, and the sulphur is transformed into an alkaline sulphostannate, chlorine, bromine and iodine give corresponding soda salts.

**Action of Chloroform on Beta-Naphtol.** By G. ROUSSEAU. (No. 1, July 3d, 1882.) By the action of chloroform upon beta naphtol several products are formed which can be separated into two distinct groups. 1 Insoluble in alkalies. 1 Glycol  $C_{12}H_{14}O_2$ . 2. Ether derived of the glycol  $C_{12}H_{14}O$ . 3. A mono-atomic alcohol  $C_{12}H_{14}O$ . 4. A rosin containing 96 per cent. of carbon. 2d. Soluble in alkalies.—1. Aldelyde  $C_{11}H_8O_2$ . 2. A rosin containing oxygen. During the reaction carbon monoxide is continually evolved.

**Extraction of Vanadium from the Basic Slags of "Le Creuzot."** By G. WITZ and F. OSMOND. (No. 1, July 3d, 1882.) These slags have the following composition:

Silica .....	16.50		
Alumina.....	3.80		
Lime.....	46.30		
Magnesia.....	4.00		
Protoxide of iron .....	7.07	Iron .....	5.50
"    manganese .....	5.30	Manganese.....	4.10
Sulphuric acid.....	0.63	Sulphur .....	0.25
Phosphoric " .....	13.74	Phosphorus.....	6.00
Vanadic " .....	1.92	Vanadium.....	1.08
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		99.26	

In the metallurgical establishments of Le Creuzot alone about 60,000 kilogr. of vanadium are annually produced in the slags, and it can be extracted as ammonium metavanadate or transformed

into new products especially adapted for the fabrication of aniline black.

**Basic Salts of Manganese.** By A. GORGEN. (No. 2, July 10th, 1882.)

**Action of Bromine upon Quinolein and Pyridin.** By E. GRIMAUN. (No. 2, July 10th, 1882.) By the action of bromine upon quinolein a bromide  $C_9H_9NB'$  is produced which is very instable and easily converted into  $C_9H_9NB'$ , H Br. Similar products are obtained by the action of bromine upon pyridin.

**Upon the Presence of Glycol in Wine.** By A. HENNINGER. (No. 2, July 10th, 1882.) Glycol is one of the products formed during the vinous fermentation.

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C	51
H	7.2
N	15.4

This white precipitate transforms meat into peptone.

**Two New Antiseptics.** By G. LEBON. (No. 3, July 11th, 1882.) Calcium and sodium, Glyceroborates.

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# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Room 1, University Building, N. Y.*

The last regular meeting was held Friday, Nov. 3d.

The meeting was called to order at 8:40 p. m., Prof. A. R. Leeds in the chair.

A quorum not being present, the first paper of the evening, "On the action of Phthalic anhydride, upon Gallic acid," by Mr. James H. Stebbins, Jr., was then read, at the conclusion of which Dr. Prochazka remarked that he had produced galleine by the above method, over a year ago.

After which the second paper, "On the preparation of Asbestos, for filtering purposes," by Mr. Casamajor followed.

A quorum being now present, the minutes of previous meeting were read, and approved.

The minutes of the Board of Directors were then read, and after a correction, approved.

Dr. Doremus then stated on behalf of the Committee on Papers and Publications, that the October number of our journal is now in print, and will probably be issued next week.

He further laid before the society the necessity for members to send in more original MS., as thereon depends the success of our journal.

The committee on Endowment Fund had no report.

The following gentlemen were then elected :

Cuthbert P. Conrad, Active Member.	{ J. W. Mallett, E. Waller, James H. Stebbins, Jr.
Edwin V. Wilmowski, Regular Member.	{ James H. Stebbins, Jr., Wm. Rupp, Dr. Grothe.
Alfred L. Beebe, Regular Member.	{ J. B. Macintosh, A. L. Colby, A. H. Elliott.
John Cawley, Regular Member.	{ James H. Stebbins, Jr., A. H. Elliott, P. Casamajor.
N. Hathaway, Regular Member.	{ E. Waller, Wm. Rupp, C E. Munsell.

Geo. E. Leguin, Associate Member. { Nelson H. Darton,  
James H. Stebbins, Jr.,  
Wm. Rupp.

John Grey Macfarlan. { P. Casamajor,  
James H. Stebbins, Jr.,  
A. H. Elliott.

The following gentleman was nominated as a Regular Member :

Percy Neymann. { E. Waller,  
A. H. Elliott, } Proposed by.  
A. P. Hallock,

The election of candidates for the Committee on Nominations was now in order.

The following gentlemen received the largest number of ballots :

1. Arthur H. Elliott.
2. E. Waller.
3. James H. Stebbins, Jr.
4. A. R. Ledoux.
5. Wm. Habirshaw.
6. P. Casamajor.
7. J. P. Battershall.
8. A. P. Hallock.
9. O. Grothe.

Three names out of the nine were then drawn from a hat by the Chairman.

The members of the Nominating Committee are therefore, Wm. Habirshaw, J. P. Battershall, P. Casamajor.

There being no further business, the meeting adjourned.

JAMES H. STEBBINS, Jr.,  
Recording Secretary.

## ON THE ACTION OF PHTHALIC ANHYDRIDE, UPON GALLIC ACID.

BY JAMES H. STEBBINS JR., A.B., F.C. S.

In 1871 Baeyer observed, while heating a mixture of phthalic acid, and pyrogallie acid, the formation of a coloring matter,\* which he termed "galleine."

At first Baeyer, believed that the formation of this dyestuff, was due to an oxydation of pyrogallie acid, and that the phthalic acid was not essential for its formation, as it seemed that the same dyestuff

\* Ber. d. d. chem. Gesell. IV., p. 455, 555, 603.



was formed by heating pyrogallic acid, with other organic acids, or with benzaldehyde.

Further investigation, however, soon showed, that the phthalic acid, as well as the other acids, on being heated with pyrogallic acid, enter the molecule of the resulting compound, and that not only does pyrogallic acid act in this way, but also phenoles on the one hand, and many organic acids on the other, combine under elimination of water, when the mixture, is either heated alone with glycerine, or with sulphuric acid.

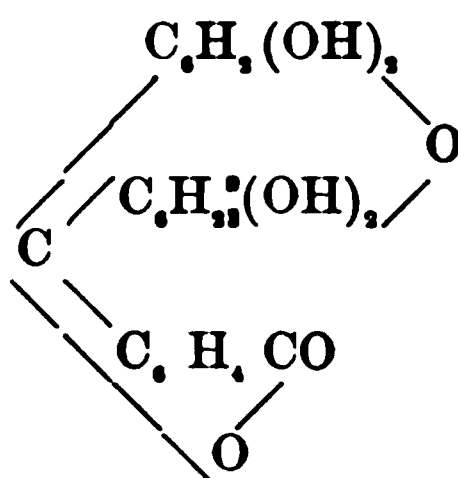
The constitution of the compounds (which he called phthaleines) was in later years explained by Baeyer, and his scholars.\*

Baeyer in his last paper on this subject (2) assumes that the formula of galleine may be represented by



which on being heated to  $180^{\circ}\text{C}$  loses water, and passes over into a compound closely resembling fluoresceine, which he called galline. ( $C_{30}H_{12}O_7$ )

In order not to make this subject too tedious, I will omit the many deductions, as to the correct formula of galleine, and will use Baeyer's formula hereafter, viz.:

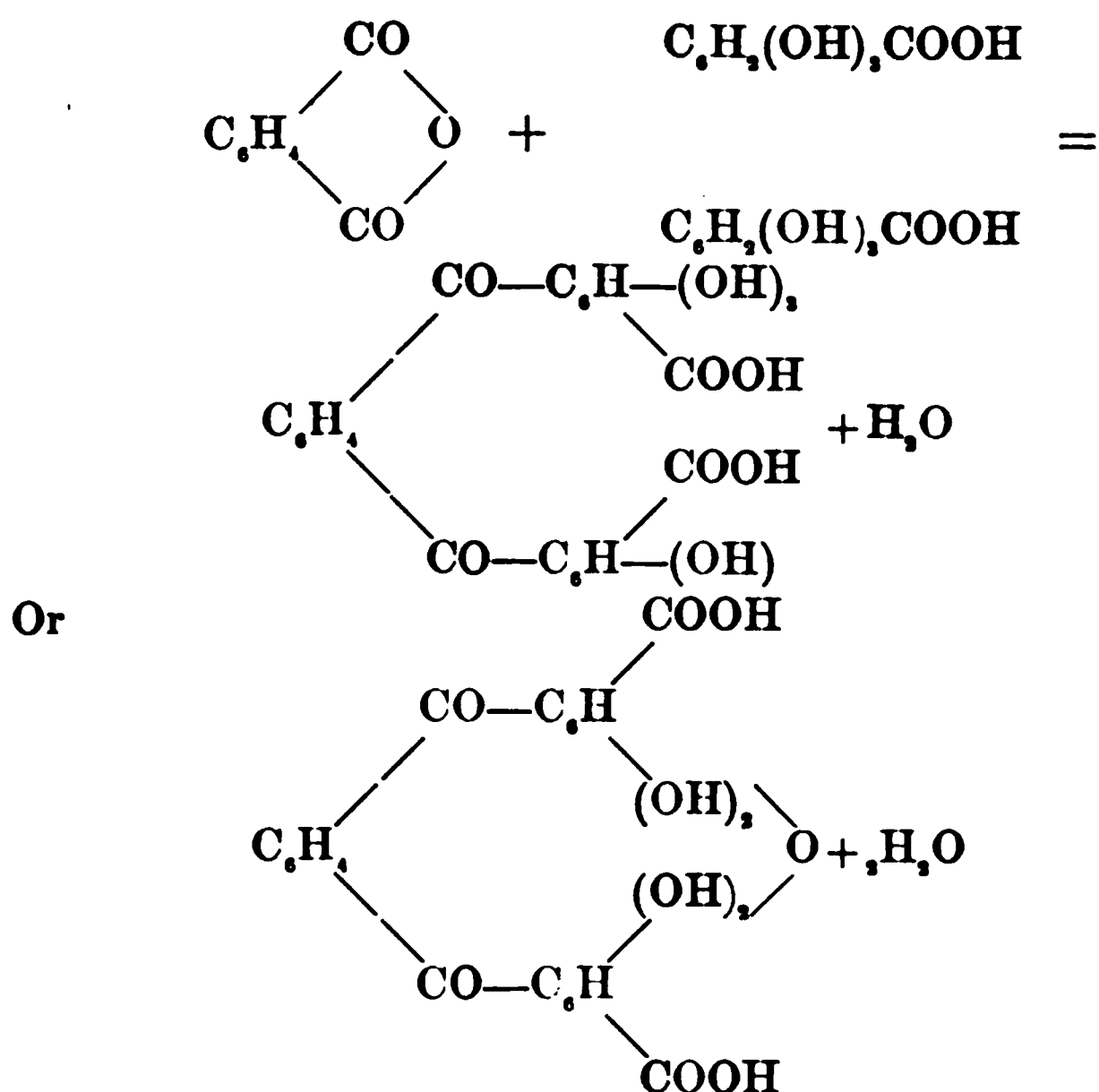


While reading over some of Baeyer's interesting work on this subject, it occurred to me, that possibly phthalic-anhydride, and gallic acid, might be combined, to form a compound of similar nature to galleine.

According to Baeyer's theory, phthalic-anhydride and gallic acid should unite, under elimination of water, to form a substance, which might possibly be represented by the following formulæ :

\* Ann. Chem. and Pharm. 183,1; 202 p. 36.

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But, the reaction did not work according to my expectations, as will be seen from the following:

10 grs. phthalic-anhydride, and 20 grs. gallic acid, were thoroughly mixed and heated at first, (on the sand bath) to 195°C. At this temperature no reaction took place. So the heat was gradually raised to 220°C., at which temperature the mixture melted, under evolution of a gas, which on examination was found to be CO<sub>2</sub>. The melt then darkened in color, and finally turned red brown.

The whole time of heating was about two hours.

The melt was then allowed to cool, dissolved in boiling alcohol, and the solution so obtained poured into cold water.

A copious fine brown precipitate is thus formed. This was collected on a filter, washed with water, dried, and finally recrystallized from alcohol

A combustion of the above dried at 120°C. gave the following figures:

	FOUND.
Carbon.....	61.50 per cent.
Hydrogen.....	3.50 per cent.
	THEORY.
Carbon.....	62.82 per cent.
Hydrogen.....	3.63 per cent.

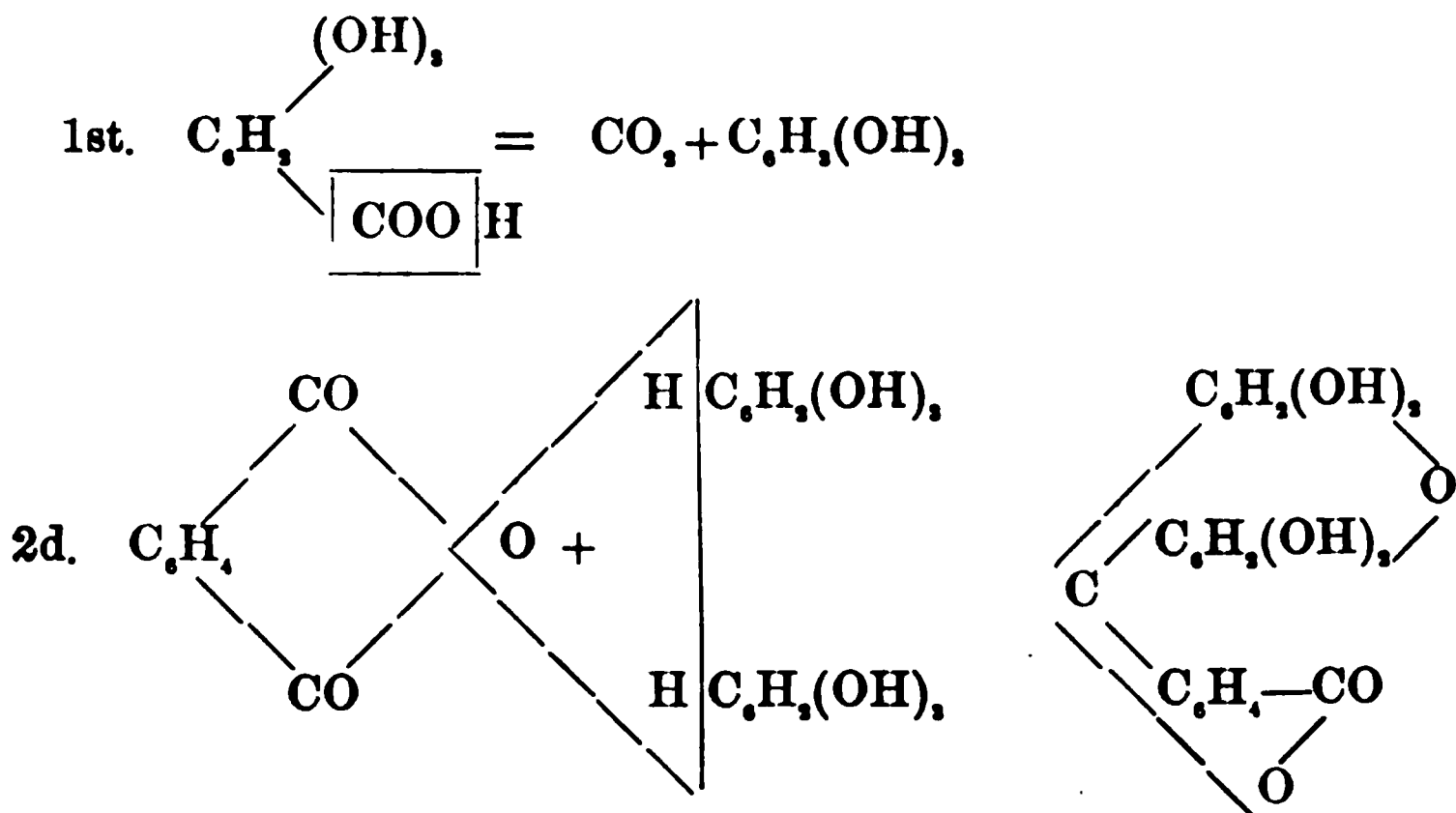
These figures, beyond a doubt, lead to the formula of galleine, and in fact the body analysed closely resembles the latter in every respect.

It dissolves in alcohol, with a fine cherry-red color. Is sparingly soluble in hot water, and dyes mordanted cotton, of the same shades as galleine. It is likewise soluble in caustic soda solution, with a fine blue color, and finally, it forms with sulphuric acid, a condensation product, closely resembling coeruleine.

The formation of galleine from phthalic-anhydride, and gallic acid may, I think, be explained as follows.

1st. The gallic acid, on being heated to 220°C. splits up into carbonic acid, and pyrogallic acid.

2nd. The pyrogallic acid on being formed then unites with the phthalic acid to produce galleine. Thus:



Finally, to make sure, that the compound under examination was really galleine, I treated a small amount of it with acetic anhydride, and thus obtained the well known tetra acetyl galleine, as may be seen from the following figures:

FOUND.		
I.		II.
Carbon .....	64.09 per cent.	64.09 per cent.
Hydrogen .....	4.71 per cent	4.27 per cent.
THEORY.		
$\text{C}_{20}\text{H}_8(\text{OCOCH}_3)_4$		
Carbon .....	63.15 per cent.	
Hydrogen .....	3.75 per cent.	

The yield of galleine by this process is quite large, and it promises, I think, to be a good improvement in the manufacture of galleine, on account of the greater cheapness of the raw materials.

N.B. After this paper had been read before the American Chemical Society, Dr. Prochazka arose and stated that he had obtained the same results as myself, about a year previous, and I therefore cannot claim any originality in these experiments, but publish them merely for the benefit of the public, as Dr. Prochazka's experiments have remained hid under a bushel.

JAMES H. STEBBINS, JR.

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### NOTE ON ASBESTOS FILTERS.

BY P. CASAMAJOR.

An extensive dealer in wares used by chemists, lately informed me that he had many inquiries concerning asbestos for filtering liquids in chemical analysis. Some chemists complain that they cannot get clear solutions through asbestos, while others, who obtain clear solutions, find that their liquids filter altogether too slowly.

Allow me to recall that the method of making asbestos filters, by pouring a thin paste of this material over a perforated platinum disc, was first proposed by me in 1875,\* but I neglected to give directions concerning the preparation of asbestos, to make it fit for filtering liquids in chemical analysis. It now appears, however, that such directions would be found useful, and I propose to repair the omission and give the necessary details.

The kind of asbestos to use is a matter of some importance. I have tried three kinds, which are sold by dealers in New York, as the *Canadian*, the *Italian*, and the *Australian*. This last is less flexible than the other two, and consequently the fibres do not felt together and pack as closely on the perforated plate. Hence, liquids filter more rapidly, and the Australian is, on this account, preferable to the other two kinds. I am informed that the Canadian asbestos is the most soluble in acids, but I have not verified the assertion.

Whatever may be the kind of asbestos used, the following is a

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\* See *American Chemist*, V., p. 44. *Chemical News*, XXXII., p. 46.

process for obtaining, with little trouble, a quantity of the pulp in a fit state for filtration :

A coarse brass sieve is placed over a sheet of paper, and a handful of asbestos is rubbed pretty roughly over the sieve cloth. This breaks it up in such a way, that the smaller fragments pass through the meshes, and are deposited on the paper underneath. After a while, the portion which remains on the sieve-cloth is collected in one bundle, and rubbed again in the same manner, and the operation is repeated until a sufficient quantity has gone through. In a few minutes, enough of the material is obtained to last for months.

As to the coarseness of mesh to use, I may say that I have used No. 10 sieve (ten openings to the inch) with satisfactory results. The sieve is best placed bottom up, so as to leave plenty of room under the cloth.

The next operation is to free the sifted material from dust and from the finest particles. This is easily accomplished by placing the asbestos, as obtained above, over another sieve of finer mesh (about No. 25 or No. 30), and stirring it while water is poured over the sieve. The first water which passes through is quite milky, but it gradually becomes clearer as the washing is continued. The washed asbestos is then put in a beaker glass, and boiled for about half an hour with strong hydrochloric acid (about 1 part of fuming HCl. to 4 parts of water).

The pulp, after this treatment, is poured over a perforated platinum plate placed in a funnel,\* and washed with distilled water until no acidity is shown by litmus paper. The pulp is then taken out of the funnel and strongly heated in a platinum dish. After letting it cool sufficiently, it may be placed in a wide-mouth bottle for future use.

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\* A perforated platinum disc, having a stout platinum wire soldered with gold in the centre of the disc, can be used in an ordinary conical funnel. The wire finds its place in the stem of the funnel, and keeps the perforated plate in position. (See *Chemical News*, XLVI., p. 8). A Gooch crucible of sufficient size may also be used for the same purpose.

The yield of galleine by this process is quite large, and it promises, I think, to be a good improvement in the manufacture of galleine, on account of the greater cheapness of the raw materials.

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**Action of Carbon Di-Sulphide upon Silicium.** By A. COLSON. No. 23. (June 5, 1882).

**Preliminary Note on Didymium.** By T. CLEVE. No. 23. (June 5, 1882). The differences obtained in the determinations of the atomic weight of didymium have induced the author to believe in the existence of a new element which he calls beta-didymium.

**Explosible Alloys of Zinc with the Platinum Metals.** By SAINTE-CLAIRE DEVILLE and H. DEBRAY. No. 24. (June 12, 1882).

**Separation of Gallium.** By LECOQ DE BOISBAUDRAN. No. 25. (June 19, 1882). Separation of gallium from zirconium, manganese and zinc.

**Upon Silicium.** By P. SCHUTZENBERGER and A. COLSON. No. 26. (June 26, 1882).

**On the Compound  $\text{NH}_3$ .** By M. COMBES. No. 26. June 26, 1882). According to the author, the compound  $\text{NH}_3$  has not been obtained.  $\text{NH}_3$  and carbonic acid had been formed in the reaction alluded to by M. Maumene.

**On Didymium.** By B. BRAUNER. No. 26. (June 26, 1882). Ordinary didymium is a mixture of three elements—didymium, beta-didymium and perhaps samarium.

**Action of Peroxide of Hydrogen upon the Coloring Matter of Blood and upon Hematosin.** By A. BECHAMP. No. 26. (June 26, 1882). Description of the action of peroxide of hydrogen upon hemoglobin and hematosin and upon the white matter of hemoglobin.

**Electrolysis of Hydrogen Dioxide.** By M. BERTHELOT. (No. 1, July 3d, 1882.)

**Remarks on Didymium.** By P. T. CLEVES. (No. 1, July 3d, 1882.) In a preliminary note the author suggested the existence of a new element which he named Beta-Didymium, but further experiments have shown the non-existence of this new element.

**Action of Hydrogen Sulphide upon Nickel Chloride.** By H. BAUBIGNY. (No. 1, July 3d, 1882.) The transformation into sulphides of some metallic chlorides is more difficult than the transformation of the corresponding sulphates, under the same conditions,



by the action of hydrogen sulphide. Equivalent weights of sulphate and chloride of nickel being taken and submitted under the same conditions to the action of hydrogen sulphide, it has been observed that the conversion of the sulphate into sulphide was quicker than for the chloride.

**Isomery of Cuprous Sulphites.** By M. ETARD. (No. 1, July 3d, 1882.)

**Reduction of Some Silver Ores by Hydrogen and the Wet Way.** By P. LAUR. (No. 1, July 3d, 1882.) The silver ore, sulphide, chloride, iodide or bromide in fine powder is placed in a cast iron pan with a weak alkaline solution (1 part of soda for 100 of water) and an amalgam of 3 parts of tin for 100 of mercury and submitted to ebullition, nascent hydrogen decomposes the argentiferous compounds, the silver forms an amalgams, and the sulphur is transformed into an alkaline sulphostannate, chlorine, bromine and iodine give corresponding soda salts.

**Action of Chloroform on Beta-Naphtol.** By G. ROUSSEAU. (No. 1, July 3d, 1882.) By the action of chloroform upon beta naphtol several products are formed which can be separated into two distinct groups. 1 Insoluble in alkalies. 1 Glycol  $C_{12}H_{14}O_2$ . 2. Ether derived of the glycol  $C_{12}H_{14}O$ . 3. A mono-atomic alcohol  $C_{12}H_{14}O$ . 4. A rosin containing 96 per cent. of carbon. 2d. Soluble in alkalies.—1. Aldelyde  $C_{11}H_8O_2$ . 2. A rosin containing oxygen. During the reaction carbon monoxide is continually evolved.

**Extraction of Vanadium from the Basic Slags of "Le Creuzot."** By G. WITZ and F. OSMOND. (No. 1, July 3d, 1882.) These slags have the following composition:

Silica .....	16.50		
Alumina.....	3.80		
Lime.....	46.30		
Magnesia.....	4.00		
Protoxide of iron.....	7.07	Iron .....	5.50
"    manganese	5.30	Manganese.....	4.10
Sulphuric acid.....	0.63	Sulphur .....	0.25
Phosphoric " .....	13.74	Phosphorus.....	6.00
Vanadic " .....	1.92	Vanadium.....	1.08
<hr/>			
99.26			

In the metallurgical establishments of Le Creuzot alone about 60,000 kilogr. of vanadium are annually produced in the slags, and it can be extracted as ammonium metavanadate or transformed

into new products especially adapted for the fabrication of aniline black.

**Basic Salts of Manganese.** By A. GORGEN. (No. 2, July 10th, 1882.)

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7. J. P. Battershall.
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9. O. Grothe.

Three names out of the nine were then drawn from a hat by the Chairman.

The members of the Nominating Committee are therefore,  
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There being no further business, the meeting adjourned.

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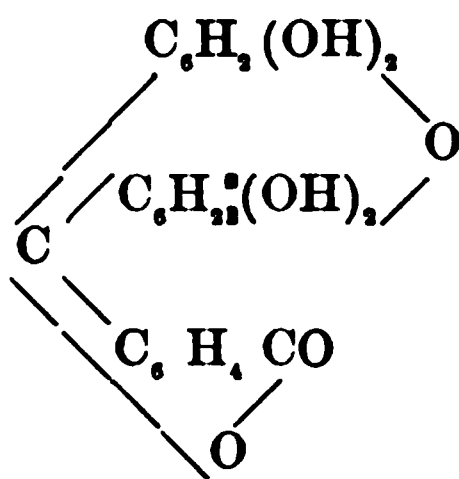
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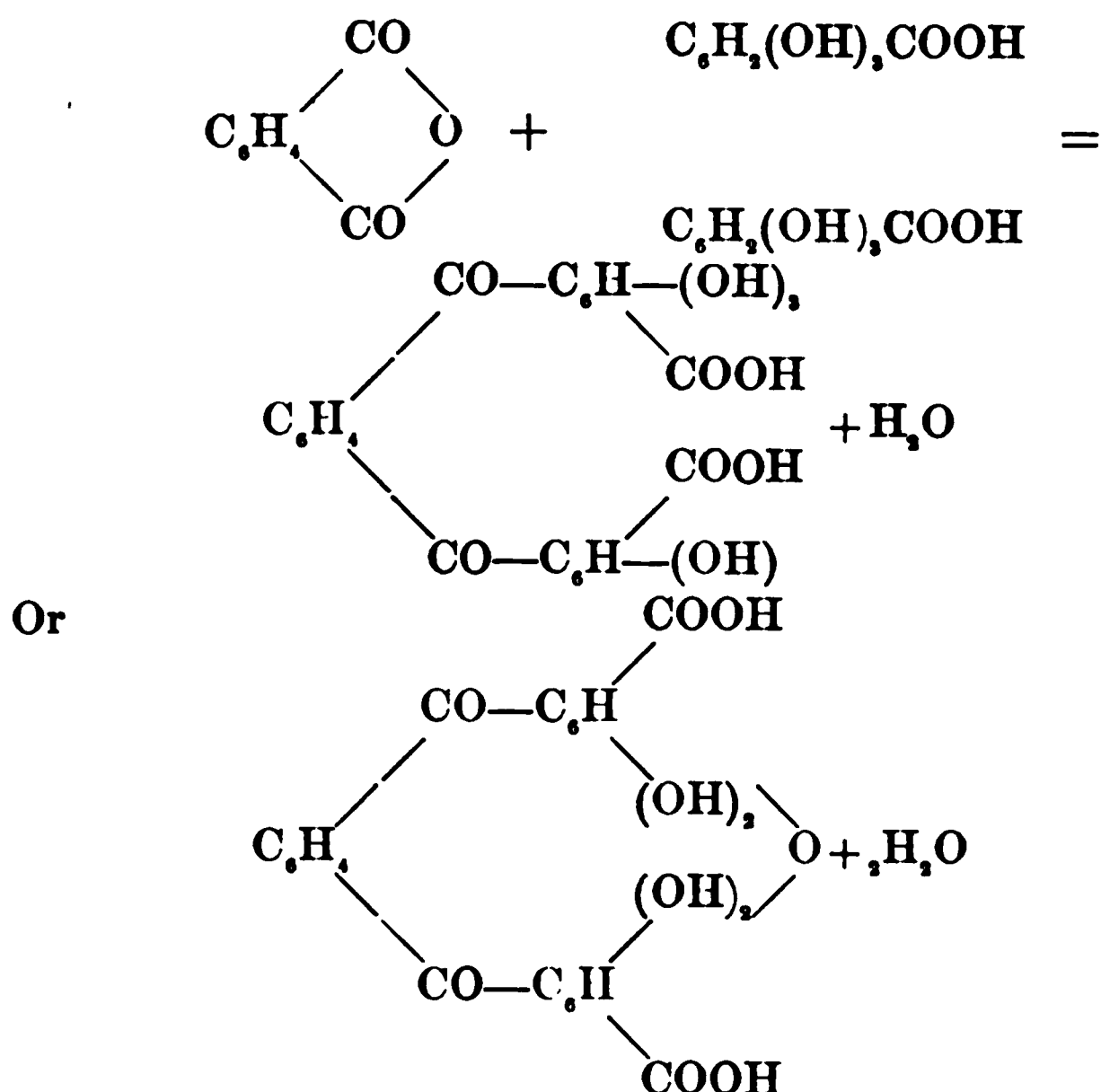


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But, the reaction did not work according to my expectations, as will be seen from the following:

10 grs. phthalic-anhydride, and 20 grs. gallic acid, were thoroughly mixed and heated at first, (on the sand bath) to 195°C. At this temperature no reaction took place. So the heat was gradually raised to 220°C., at which temperature the mixture melted, under evolution of a gas, which on examination was found to be CO<sub>2</sub>. The melt then darkened in color, and finally turned red brown.

The whole time of heating was about two hours.

The melt was then allowed to cool, dissolved in boiling alcohol, and the solution so obtained poured into cold water.

A copious fine brown precipitate is thus formed. This was collected on a filter, washed with water, dried, and finally recrystallized from alcohol

A combustion of the above dried at 120°C. gave the following figures:

	FOUND.
Carbon.....	61.50 per cent.
Hydrogen.....	3.50 per cent.
	THEORY.
Carbon.....	62.82 per cent.
Hydrogen.....	3.63 per cent.

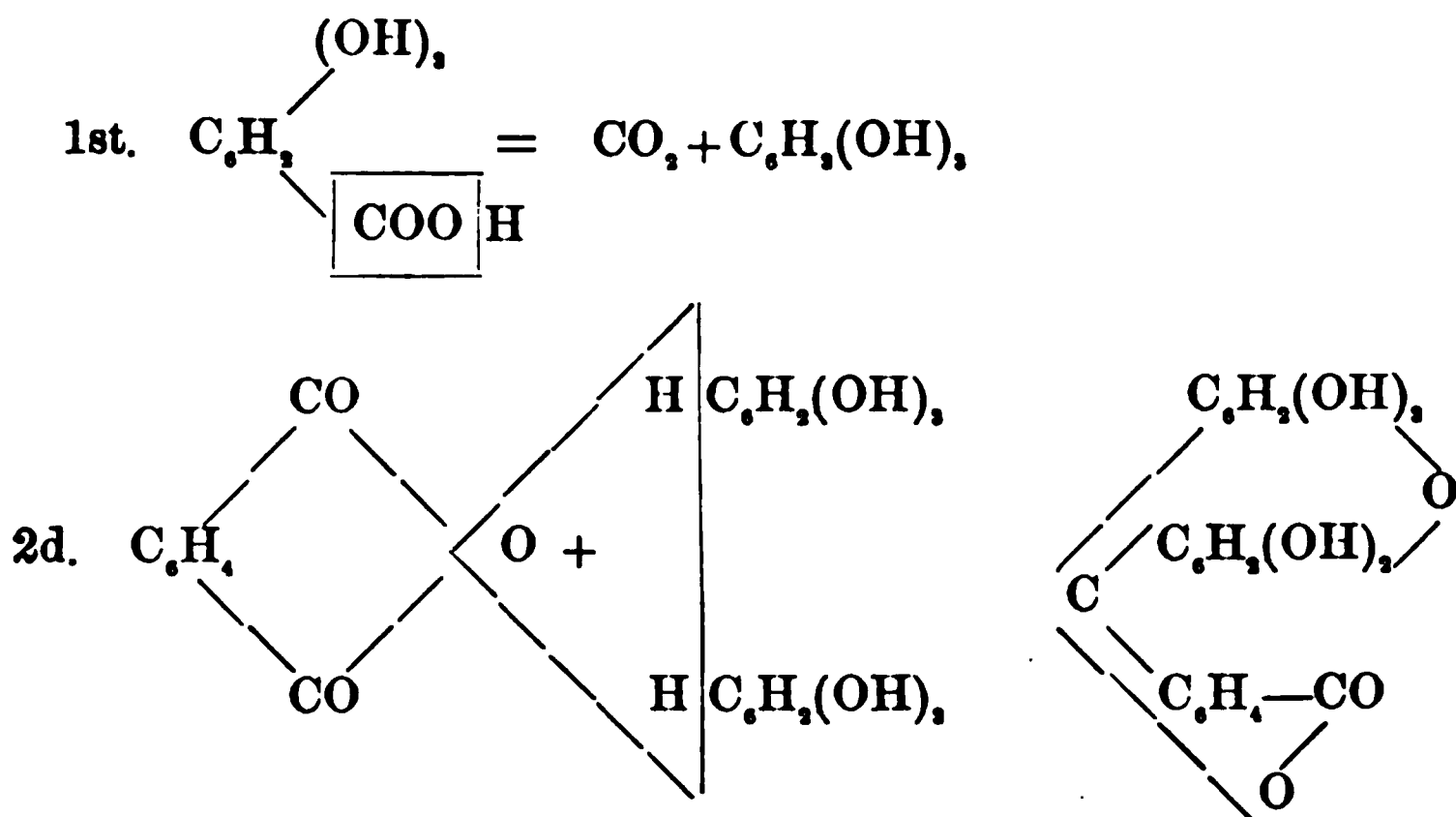
These figures, beyond a doubt, lead to the formula of galleine, and in fact the body analysed closely resembles the latter in every respect.

It dissolves in alcohol, with a fine cherry-red color. Is sparingly soluble in hot water, and dyes mordanted cotton, of the same shades as galleine. It is likewise soluble in caustic soda solution, with a fine blue color, and finally, it forms with sulphuric acid, a condensation product, closely resembling coeruleine.

The formation of galleine from phthalic-anhydride, and gallic acid may, I think, be explained as follows.

1st. The gallic acid, on being heated to 220°C. splits up into carbonic acid, and pyrogallic acid.

2nd. The pyrogallic acid on being formed then unites with the phthalic acid to produce galleine. Thus:



Finally, to make sure, that the compound under examination was really galleine, I treated a small amount of it with acetic anhydride, and thus obtained the well known tetra acetyl galleine, as may be seen from the following figures:

FOUND.		
I.		II.
Carbon .....	64.09 per cent.	64.09 per cent.
Hydrogen .....	4.71 per cent	4.27 per cent.
THEORY.		
$\text{C}_{20} \text{H}_8 (\text{OCOCH}_3)_4$		
Carbon .....	63.15 per cent.	
Hydrogen .....	3.75 per cent.	

The yield of galleine by this process is quite large, and it promises, I think, to be a good improvement in the manufacture of galleine, on account of the greater cheapness of the raw materials.

N.B. After this paper had been read before the American Chemical Society, Dr. Prochazka arose and stated that he had obtained the same results as myself, about a year previous, and I therefore cannot claim any originality in these experiments, but publish them merely for the benefit of the public, as Dr. Prochazka's experiments have remained hid under a bushel.

JAMES H. STEBBINS, JR.

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### NOTE ON ASBESTOS FILTERS.

BY P. CASAMAJOR.

An extensive dealer in wares used by chemists, lately informed me that he had many inquiries concerning asbestos for filtering liquids in chemical analysis. Some chemists complain that they cannot get clear solutions through asbestos, while others, who obtain clear solutions, find that their liquids filter altogether too slowly.

Allow me to recall that the method of making asbestos filters, by pouring a thin paste of this material over a perforated platinum disc, was first proposed by me in 1875,\* but I neglected to give directions concerning the preparation of asbestos, to make it fit for filtering liquids in chemical analysis. It now appears, however, that such directions would be found useful, and I propose to repair the omission and give the necessary details.

The kind of asbestos to use is a matter of some importance. I have tried three kinds, which are sold by dealers in New York, as the *Canadian*, the *Italian*, and the *Australian*. This last is less flexible than the other two, and consequently the fibres do not felt together and pack as closely on the perforated plate. Hence, liquids filter more rapidly, and the Australian is, on this account, preferable to the other two kinds. I am informed that the Canadian asbestos is the most soluble in acids, but I have not verified the assertion.

Whatever may be the kind of asbestos used, the following is a

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\* See *American Chemist*, V., p. 44. *Chemical News*, XXXII., p. 46.



process for obtaining, with little trouble, a quantity of the pulp in a fit state for filtration :

A coarse brass sieve is placed over a sheet of paper, and a handful of asbestos is rubbed pretty roughly over the sieve cloth. This breaks it up in such a way, that the smaller fragments pass through the meshes, and are deposited on the paper underneath. After a while, the portion which remains on the sieve-cloth is collected in one bundle, and rubbed again in the same manner, and the operation is repeated until a sufficient quantity has gone through. In a few minutes, enough of the material is obtained to last for months.

As to the coarseness of mesh to use, I may say that I have used No. 10 sieve (ten openings to the inch) with satisfactory results. The sieve is best placed bottom up, so as to leave plenty of room under the cloth.

The next operation is to free the sifted material from dust and from the finest particles. This is easily accomplished by placing the asbestos, as obtained above, over another sieve of finer mesh (about No. 25 or No. 30), and stirring it while water is poured over the sieve. The first water which passes through is quite milky, but it gradually becomes clearer as the washing is continued. The washed asbestos is then put in a beaker glass, and boiled for about half an hour with strong hydrochloric acid (about 1 part of fuming HCl. to 4 parts of water).

The pulp, after this treatment, is poured over a perforated platinum plate placed in a funnel,\* and washed with distilled water until no acidity is shown by litmus paper. The pulp is then taken out of the funnel and strongly heated in a platinum dish. After letting it cool sufficiently, it may be placed in a wide-mouth bottle for future use.

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\* A perforated platinum disc, having a stout platinum wire soldered with gold in the centre of the disc, can be used in an ordinary conical funnel. The wire finds its place in the stem of the funnel, and keeps the perforated plate in position. (See *Chemical News*, XLVI., p. 8). A Gooch crucible of sufficient size may also be used for the same purpose.

## FAT EXTRACTING APPARATUS.

T. O'CONOR SLOANE, PH.D.

In many processes of analysis a continuously working apparatus for extraction with alcohol or ether is useful. The extraction of chinchona barks, for separation of the alkaloids may be instanced. In their analysis it is best to work upon large quantities, and a long treatment with the solvent is necessary. Any attempt to thoroughly extract a large quantity by percolation and decantation would be tedious and probably imperfect. A well arranged fat-extractor is particularly available for analysis of this type.

The cut, accompanying this, shows a very efficient and cheap form of fat extractor.

*A* represents a glass receptacle in which the substance weighed out for analysis is placed. It will be recognized as one of the pieces used in the apparatus for extraction of soils. The bottom may be closed by a piece of sponge, and broken glass placed on this to a depth of two or three centimetres. The substance to be extracted rests on the broken glass.

*B* represents a glass flask. The alcohol or other extracting fluid is boiled in it, and the vapor is condensed in the condenser shown above it.

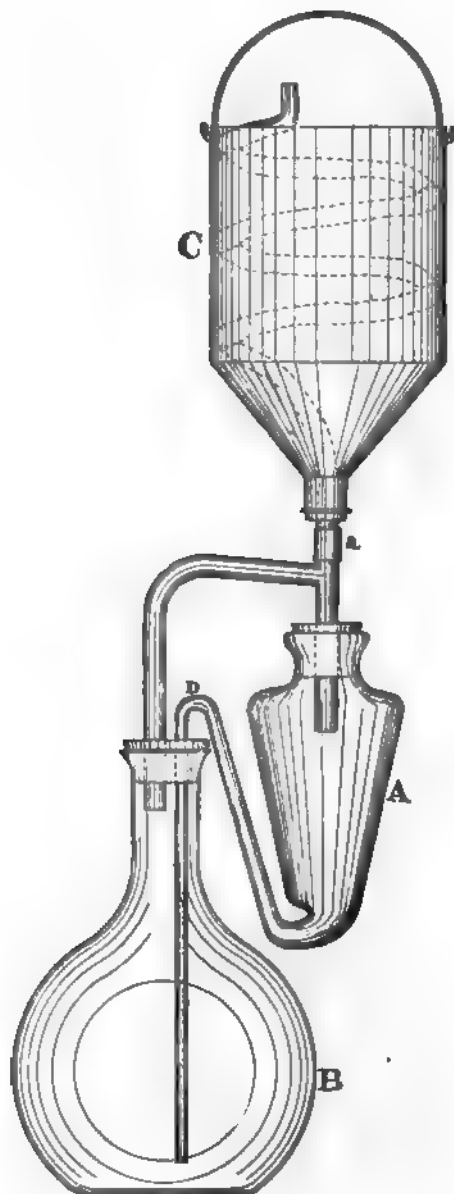
*C* is the condenser in question. It may be made with block tin pipe for the coil, as shown by the dotted lines, and a varnish can may serve for the case. If the varnish should be used, the bottom must be cut off, and the handle transferred from top to bottom as shown. The tin coil pipe passes through a perforated cork in the neck.

The other connections may be made of glass tubing, joined to the tin tube at *a* by a rubber tube.

The condenser is to be filled with water. While a continuous supply is desirable, it is not necessary, provided the condenser be large enough. In that case the water may be changed every hour, the warm water being syphoned off without dismounting the apparatus.

The operation is simple. The fluid in the flask *B* is boiled. Its vapor rises, condenses in the condenser *C*, and drops down into the receiver *A*. When sufficient has accumulated to rise above the bend *D* it syphons into the flask, and the fluid is rapidly drawn out of the receiver, until by the admission of air at the bottom of the receiver

the syphon is broken. A new accumulation of condensed fluid then takes place, which is soon syphoned off. Thus a continuous series



of rapid percolations is produced, exhausting the substance in a most effective manner.

With a boiling flask of a litre capacity, a receiver of four hundred c. c. capacity, and a condenser of five litres capacity, the extracting fluid being alcohol, the syphonings should follow each other at intervals of about twenty minutes.

The nearly horizontal portion of glass tube to the left of *A* should pitch downwards towards the receiver. This is very important, as otherwise the condensed fluid would partly run back into the flask.

Where alcohol or ether is used, a water bath should be employed to boil it.

All the tube connections should be as large as possible to insure rapidity of working.

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## ON BISULPHIDE OF CARBON.

By L. H. FRIEDBURG, PH. D.

Several years ago I published some notes on bisulphide of carbon\* to which I shall add to day a few more observations. Then and there I showed how to clean the bisulphide by means of fuming nitric acid, and that the vapors of nitrous acid, of nitrogen dioxyde, of sulphurous acid, etc., etc., were taken up and invariably retained by the bisulphide. Dry bisulphide of carbon serves as a very good conveyance for the reaction of such gases and vapors in a dry state on each other and on other substances. The only disagreeable feature in this regard is that carbon bisulphide in most cases also enters the reaction, forming very undesirable products, and sometimes, only such, sulphur containing, products are formed, in any notable quantity. The following reactions are the only three I wish to mention, as they may prove germs for further investigations.

1. Bisulphide of carbon charged with the vapors of nitrogen dioxyde and then mixed with pure benzol, forms amongst other products large, broad crystals of dinitro-benzol, melting at  $+86^{\circ}\text{C}$ . These crystals are formed after standing a considerable time, and after the partial evaporation of the mixed liquids at summer heat.

2. I think that great interest is attached to the reaction of the aforesaid liquids in direct sunlight. The brown vapors begin to disappear without escaping from the narrow neck of the very large flask, in which such experiments take place, and in proportion as they disappear, small white crystals begin to cover the sides of the

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\* Berichte d.d. ch. Ges. VIII., 1616.

flask within. This covering principally takes place above the edge of the liquid on the bare glass. The crystals could not be analyzed, because they decomposed when brought in contact with air, yielding then  $\text{NO}_2$  and benzol. It is not entirely out of the way to suppose, that in this case addition products formed, analogous to benzol-hexachloride  $= \text{C}_6\text{H}_6\text{Cl}_6$ , so that the white crystals in this case, might be either  $\text{C}_6\text{H}_6(\text{NO}_2)_6$  or  $\text{C}_6\text{H}_6(\text{NO}_2)_3$ , which of course needs further investigation.

3. A very pretty reaction takes place, when bisulphide of carbon charged with dry sulphurous acid gas, and the same medium charged with nitrogen dioxyde (which was not free from nitrous acid) are brought together. This reaction might be used as a lecture experiment. Keeping the vessels cool and dry (I generally use a spacious beaker) white crystals very readily form in considerable quantity, which, in fact, are nothing but lead chamber crystals. This reaction treated analytically may some day throw new light on the formation and composition of lead chamber crystals.

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The bisulphide of carbon cleaned by means of fuming nitric acid is the only chemically pure I came across, and I therefore proceeded to determine its specific gravity as well as boiling point, without finding though any differences from former determinations.

The spec.-gravity at  $+15^\circ.2$  C. is 1,266 and the boiling point is  $+47.4$  C., at 0.760m pressure.

The pure bisulphide shows materially no other so-called physical properties than those known heretofore.

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Finally I want to state, that in treating raw bisulphide of carbon, coming from the retorts of manufacture, with fuming nitric acid, I could invariably detect mononitrobenzol in the residue of evaporation, which leads me to believe that amongst the numerous products formed in the red hot retorts, particularly if the charcoal was not dry enough, there is also benzol.

2. The purifying influence of a non-colored and inodorous fat, for instance such as the oil of African palm kernels\* good as it is for the bisulphide of carbon, becomes a nuisance, when this latter is used as a means of extraction of the former. I advise, therefore, from long experience, all those who extract fats (particularly if it is for soap manufacturing) by means of  $\text{CS}_2$ , never to use an excess of this latter, and never to let a new mixture of oil and bisulphide run into the still in which oil already freed from bisulphide is retained. This latter will else be rendered impure.

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\* See foot note page 252.

NOTE ON THE ACTION OF ALCOHOLIC POTASH ON  
HEPTYLENE BROMIDE.

F. P. VENABLE, PH. D.,

Having on hand a small supply of heptylene bromide, described in this journal (IV. 22), the action of an alcoholic solution of potassium hydrate was tried upon it with a view to the formation of a hydrocarbon with two less carbon atoms a heptene isomeric with the oenanthylidene of Limpricht (Liebig's Annalen 103.81) and the tetra methylallylene of Henry (Ber. Chem. Ges. 8. 400). These two bodies were formed by the action of alcoholic potash on the chlorides. It was interesting then to see if the reaction with this bromide would follow the same course.

From seven to eight grammes of heptylene bromide were placed in a flask and an excess of a strong alcoholic solution of caustic potash added (2 mol. KOH to 1 mol.  $C_7H_{13}Br$ , being the proportion required by formula). To condense and return the alcohol to the flask it was connected with an upright condenser. The reaction commenced immediately in the cold—potassium bromide beginning to deposit—but it went on very slowly without the aid of heat. The whole was accordingly heated 4–6 hours on a water bath, the potassium salt forming a thick deposit on the sides of the flask and the liquid, at first colorless, darkening to a reddish black. At the expiration of this time no further precipitation of potassium bromide was observable on adding fresh alcoholic potash. The oily liquid was thoroughly washed with water, separating in a lighter layer at the top of the separating funnel, was dried over calcium chloride and finally fractioned under  $100^{\circ}C$ ., the amount coming over was small. The thermometer then rose rapidly to  $150^{\circ}C$ . then slowly to  $160^{\circ}C$ . Between these two degrees nearly the whole product came over, leaving a dirty, thick mass behind in the fractionating flask. On redistilling the fraction from  $150^{\circ}$ – $160^{\circ}C$ , a small portion of a constant-boiling product was obtained—boiling at  $156^{\circ}$ – $158^{\circ}C$ . This was immediately sealed in a tube and set aside for analysis. On examining it qualitatively, bromine was found present in considerable amount so that clearly some brominated compound instead of the expected  $C_7H_{12}$ , had been formed by the reaction. Seeing that Bruylaut (Ber. Chem. Ges. 8. 409) had prepared a monobromœnanthylene by the action of alcoholic potash on oenanthylene bromide (heated together two days in closed tubes), a

bromine determination was made after the method of Carius to see if some similar body had not been found in this case. A confirmatory determination would have been made but unfortunately part of the liquid was lost.

0.1895 grm. substance gave 0.2019 grm. silver bromide.

Calculated for  $C_7H_{11}Br$ , 45.19 per cent. Br—found 45.34 per cent. Probably the first small fraction coming over somewhat under  $100^\circ$  consisted of  $C_7H_{12}$  but the quantity was too small for examination and purification. The reaction then, instead of following the formula,  $C_7H_{14}Br_2 + 2KOH = C_7H_{12} + 2KBr + 2H_2O$  was rather  $C_7H_{14}Br_2 + KOH = C_7H_{11}Br + KBr + H_2O$  in spite of the excess of alcoholic potash.

Bromheptylene— $C_7H_{11}Br$  is a colorless mobile liquid, with a pleasant though pungent aromatic odor, lighter than water and boiling at  $156^\circ$ – $158^\circ C$ . The two bodies  $C_7H_{11}Br$ , and  $C_7H_{12}$ , prepared by Bruylaut had respectively the boiling points  $165^\circ C$  and  $100^\circ C$ . This gives a difference of  $7^\circ$ – $9^\circ$  in boiling points between the derivation from the ænanthal heptane and the Pinus Sabiniana heptane. Nearly the same difference is noticed between the heptylic bromides  $C_7H_{11}Br$  from ænanthal boiling at  $178.5^\circ$  and from P. Sabiniana boiling at  $165.5^\circ$ .

University of North Carolina.

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## NOTES FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF NORTH CAROLINA—ANALYSIS OF ROCK-SALT FROM SALTVILLE, WASHINGTON CO., VA.

BY THOMAS RADCLIFFE.

A specimen of this rock-salt sent by the superintendent of the salt-works in the valley of the Holston, yielded on analysis somewhat different results from the previously published analysis (Chem News No. 1038) and in view of this and the claim made for this Virginia brine, that it exceeds in purity nearly all others of which analyses are on record, the analysis made in this laboratory is published. The specimen was brownish-red in color, with a crystalline structure and was obtained whilst deepening one of the salt-wells. This rock-salt is not mined, the brine alone being used for the manufacture of salt. The capacity of the works is at present 450,000

bushels per year, though at one time, during the late war, the yield was as high as 10,000 bushels per day.

According to this analysis the rock-salt contained:

Na Cl.....	93.05
K Cl.....	trace
CaSO <sub>4</sub> .2H <sub>2</sub> O.....	2.40
MgSO <sub>4</sub> .....	.07
Fe <sub>2</sub> O <sub>3</sub> .....	.83
SiO <sub>2</sub> .....	2.81
H <sub>2</sub> O.....	.30
	<hr/>
	99.46

An analysis of the salt as marketed gave 98.89 per cent. NaCl with a small percentage of CaSO<sub>4</sub>.2H<sub>2</sub>O and a trace of MgSO<sub>4</sub>, showing it to be a high-grade salt.

## ANALYSIS OF A DEPOSIT OF ZINC OXIDE IN A BLAST FURNACE AT LONGDALE, VA.

BY THOMAS RADCLIFFE.

As is well-known these deposits of impure zinc oxide are sometimes found in furnaces where zinc-bearing ores are used. The name cadmia is given them in Dana's mineralogy. The green flame of burning zinc is noticed at the tyrup of these furnaces and was formerly looked upon by furnace-men as indicative of sulphur—especially as this burning left on substances in the near neighborhood of the tyrup a coating of zinc oxide which was yellow whilst hot. The specimen examined was sent through the courtesy of the manager of the Longdale Iron Co's. furnaces. According to analysis no zinc is contained in the ores used by this company, the said ores being ordinary brown hematite. Nor has zinc been found in the coke and limestone used—evidently occurring then in minute traces probably in the ore. The deposit was very large, nearly choking the mouth of the furnace. The specimen had a laminated appearance, as if deposited in layers, and was greenish-brown in color. It was quite hard, breaking in thin plates, like shale, in the



direction of the lamination and the specific gravity was 5.0405 (temp. of water 16°C).

The analysis gave:

ZnO.....	93.34
PbO.....	2.37
Fe (metallic).....	1.99
CaCO <sub>3</sub> .....	1.01
C.....	.20
SiO <sub>2</sub> .....	1.11
	<hr/>
	100.02

## ABSTRACTS.

Abstracts from the Journal of the Chemical Society (London), by Arthur H. Elliott, Ph. B., F. C. S.

**On Rotary Polarization of Chemical Substances under Magnetic Influence.** By W. H. PERKIN, F. R. S. (Vol. XL., p. 330).

Author used tubes 10 c. m. long, closed with glass plates; which tubes had their ends inserted a short distance into perforations in the armature of an electro-magnet, as proposed by De la Rive. Water and carbon disulphide were used as standards of comparison. The results obtained bear little or no relationship to the chemical composition of the substance examined. But by referring the observations to lengths of tubes related to each other in proportion to their molecular weights, and making the necessary corrections for density, it is found that the molecular magnetic rotary power of bodies follows more or less regularly with the chemical composition.

**A Spectroscopic Study of Chlorophyll.** By W. J. RUSSELL, Ph. D., F. R. S., and W. LAPRAIK, F. C. S. (Vol. XL., p. 334).

By making an alcohol-ether solution of leaves, the authors obtained a chlorophyll solution and studied the four least refrangible absorption bands. Authors tried the action of hydrochloric acid gas upon the spectra and observed some remarkable movements in the position of the bands. They also state that other acids produce

like changes. Carbonic acid produces no change. Application of heat produces like changes. The action of mercuric chloride, ferric chloride is also noticed. Also the alum and basic lead precipitates. By adding alkalis to the chlorophyll solution, there is a fading out of all except the least refrangible band, and the spread of this band towards the blue. Further addition of alkali divides this band into two, and still more alkali entirely eliminates all but one band.

**On the Precipitation of the Alums by Sodid Carbonate.** By EDMUND J. WILLS, D. Sc., F. R. S., and K. L. BARR, (Vol. XL., p. 343).

By a series of experiments the authors find that the precipitation of an alum by sodid carbonate takes place in three stages. First, much carbonate is added without any precipitation taking place; secondly, there is a precipitation according to a continuous law until about half the alum is thrown down; and thirdly, the precipitation proceeds according to the previous law, but with altered constants.

**On the Determination of Nitric Acid as Nitric Oxide by Means of its Reaction with Ferrous Salts. (Part II.)** By ROBERT WARRINGTON. (Vol. XL., p. 345).

This paper gives the details of a modification of Schloesing's method with ferrous chloride and hydrochloric acid, making the determinations in an atmosphere of carbon dioxide, measuring the nitric oxide by treatment with excess of oxygen in a gas analysis. It is especially applicable to the determination of nitrates in soils.

**On the Determination of Nitric Acid in Soils.** By ROBERT WARRINGTON. (Vol. XL., p. 351).

Author gives details of the manner of taking soil samples, the treatment of the samples, variations in the quantity of nitrates present in the soil resulting from different modes of drying, preparation of the watery extract, and analysis of the soil extract. Author also gives a set of tables showing the relative value of the Crum-Frankland and Schloesing methods for the determination of nitrates, giving the preference to the latter. The indigo method for the determination of nitric acid is shown to be valueless.

**Metallic Compounds containing Bivalent Hydrocarbon Radicals. (Part III).** By J. SAKURAI, F. C. S., University of Tokio, Japan. (Vol XL., p. 360).

By mixing monomeric methylene iodide with mercuric chloride,

molecule for molecule, and cohabiting with alcohol, a white crystalline body was obtained, soluble in ether, chloroform, and alcohol, but insoluble in water. It melts at  $129^{\circ}\text{C}$ . Heated with a solution of iodine in potassic iodide, it yields an oily liquid. Its formula is given as  $\text{CH}_2\text{HgICl}$  or monomeric-methylene chloride. The constitution of this body is discussed and the formula  $\text{Cl}(\text{CH}_2)\text{HgI}$  is believed to be the correct one. Incidentally, during this investigation methylene chloroide was obtained, and its boiling point is given as  $109^{\circ}\text{C}$ .

**Some Observations on the Luminous Incomplete Combustion of Ether and other Organic Bodies.** By W. H. PERKIN, F.R.S. (Vol. XL., p. 363.)

The author noticed during the evaporation of ether that a pale blue flame, visible in the dark, was floating about upon the hot sand-bath near without igniting the ether which was evaporating. The temperature at which this phenomena occurs is about  $260^{\circ}\text{C}$ ., from that to a dull red heat. A number of methods of exhibiting this phenomena at lectures are mentioned; one with a hot copper or iron ball, and another with a glass tube, which appear easy of execution. The temperature of this flame is low and will not ignite carbon disulphide. Another point about this combustion is that the quantity of carbon dioxide produced is very small. It was found that for 1.313 grm. of oxygen consumed only 0.133 grm. of carbon dioxide was found. Among other bodies that produce this effect are mentioned acetic aldehyde, the alcohols up to amyl pentane, hexane, heptane, and the hydrides up to paraffin. Benzol and homologues, phenol and cresol, give no result. The fatty acids give luminous effects; stearic acid at about  $250^{\circ}\text{C}$ . and when  $290^{\circ}\text{C}$ . is reached blue flames appear: oleic acid acts like stearic. Benzoic, cinnamic and phthalic acids give no results. Olive and linseed oils, and white wax act like oleic acid. Spermaceti shows the effect very well and is recommended for a lecture experiment. When solid paraffin was used and heated in a retort through which air was drawn 0.3816 grm. of oxygen was consumed and only 0.025 grm. of carbon dioxide obtained. Author thinks that the luminous appearance is similar to the phosphorescence obtained during the imperfect combustion of phosphorous. The light was too feeble for detailed spectroscopic analysis; but no bands were seen.

**On Some New Compounds of Hæmatein and Brazilein.**  
By J. J. HUMMEL and A. G. PERKIN. From the Dye-house of the  
Yorkshire College. (Vol. XL., p. 367.)

Authors describe a simple method of preparing crystalline hæmatein in tolerable quantity. They use the ammonio-compound of hæmatein, and by treatment with strong acetic acid obtain crystals. These crystals are microscopic rhombic plates, soluble in water, alcohol, ether and acetic acid; and alkalies dissolve them readily. Analysis gave figures agreeing with the formula  $C_{16}H_{11}O_6$  and the crystals are anhydrous.

By treating hæmatein with cold concentrated sulphuric acid and then adding acetic acid an orange colored crystalline powder is obtained, consisting of microscopic prisms; and analysis gives the formula  $C_{16}H_{11}O_6HSO_4$  or *acid isohæmatein sulphate*. It is insoluble in alcohol, ether, and benzol; but is soluble to a small extent in acetic acid. Ammonia and soda solutions dissolve it.

When hæmatin is heated in sealed tubes with hydrochloric acid, and purified by crystallization from water microscopic orange-red needles are obtained, which analysis shows to have the formula  $C_{16}H_{11}O_6Cl$ . *Isohæmatein chlorhydrin*. It dissolves easily in water; it is less soluble in alcohol; and gives a reddish violet solution with alcoholic potash. Concentrated sulphuric acid converts it into isohæmatein sulphate.

Treatment of hæmatein with hydrobromic acid in the same manner as with hydrochloric acid gives *isohæmatein monobromhydrin* as microscopic needles, having the formula  $C_{16}H_{11}O_6Br$  and resembling the chlorine compounds.

By treating isohæmatein chlorhydrin or bromhydrin with argentic hydrate, *isohæmatein* is obtained, having the formula  $C_{16}H_{11}O_6$  and isomeric with hæmatein.

*Crystalline Brazilein* is obtained from Brazil Wood extract in the same manner that hæmatein is obtained from Logwood extract. It forms thin microscopic reddish-brown rhombic plates, very slightly soluble in cold water, more so in hot. Alkalies dissolve it. Analysis gives the formula  $C_{16}H_{11}O_6H_2O$ . Treated with concentrated sulphuric acid yellow rhombic needles of *acid isobrazilein sulphate* are obtained, having the formula  $C_{16}H_{11}O_6HSO_4 + H_2O$ . It is slightly soluble in boiling glacial acetic acid, very soluble in alkalies.

By heating brazilein in sealed tubes with hydrochloric acid *isobrazilein chlorhydrin* as microscopic prisms is obtained, having the

formula  $C_{16}H_{11}O_4Cl$ . It is soluble in water and alkalies. By similar treatment with hydrobromic acid the corresponding bromine compound  $C_{16}H_{11}O_4Br$  is obtained.

The tinctorial power of the new compounds is much greater than the original hæmatein and brazilein, and much faster.

**On the Crystallization from Saturated Solutions of Certain Compound Salts.** By JOHN M. THOMSON AND W. POFFLEWELL BLOXAM. (Vol. XL p. 379).

The Paper is a continuation of the experiments published in the *Journal* May 1879, on "The Action of Isomorphous Salts in producing the Crystallization of Supersaturated Saline Solutions." In the present communication the Authors take up the case of supersaturated solutions of double salts, acting upon them with nuclei consisting of one or the other of the component salts. Of the salts experimented with are the halogen salts of mercury with potassium and ammonium, potassic alum, Lefort's salt (the double zinc-copper sulphate), microcosmic salt, double tartrates and citrates of potassium, sodium and magnesium.

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Abstracts from *Berichte der Deutschen Chemischen Gesellschaft*. By Percy Neymann, Ph.D.

**On the Behavior of Chlorosulphurous Acid with Several Metalloids and with Tin.** K. HEUMANN and P. KÆCHLIN. (Vol. XV. p. 416).

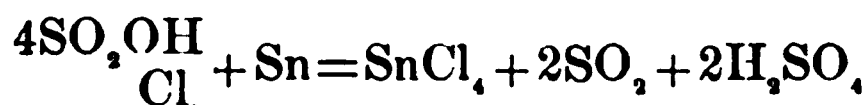
Chlorosulphurous acid  $SO_2OH.Cl$  can be regarded as an acid and also as an acid chloride. The authors experimented with chlorosulphurous acid and sulphur, phosphorous, arsenic, antimony, tin, and carbon separately.

Chlorosulphurous acid and sulphur react upon heating and a yellowish oil, found by analysis to be sulphurous chloride  $S_2Cl_2$ , was distilled. Sulphurous acid gas and hydrochloric acid escaped during the reaction.

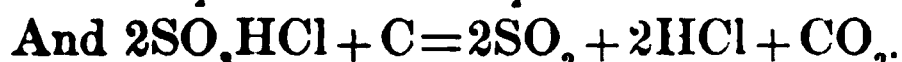
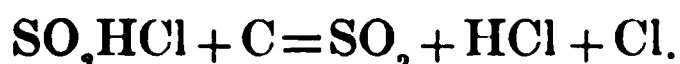
Chlorosulphurous acid and yellow phosphorus reacted best upon each other at a temperature of  $25^{\circ}$ — $30^{\circ}$  and currents of sulphurous and hydrochloric acid gases escaped. The liquid became more heated by the reaction and the experiment terminated in an explosion of the apparatus. Red phosphorous is only attacked after stronger heating and a few drops of a distillate are obtained which

seem to be phosphoroxychlorid, but this is probably the product of a second reaction.

Arsenic and antimony form their trichlorides. The reaction is characterized by leaving in both cases a non-volatile compound in the retort. The chlorosulphurous acid is most probably split up into chloride, sulphur dioxide and sulphuric acid :  $2\text{Cl. SO}_2\text{H} = 2\text{Cl} + \text{SO}_2 + \text{H}_2\text{SO}_4$ . The reaction with tin is much more complete the tetrachloride being formed according to the formula



Freshly ignited wood charcoal when strongly heated with chlorosulphurous acid generates gases which cannot be liquefied. The decomposition of chlorosulphurous acid takes place as follows :



Chlorosulphurous acid hence has a direct chloridizing action upon sulphur, phosphorous, arsenic, antimony and tin, at the same time sulphurous anhydride and sulphuric acid are produced. The latter produces secondary reactions with the respective elements and their chlorides and hence oxygen compounds of the metalloids are formed. Carbon reacts in a different manner and produces  $\text{COCO}_2\text{HCl}$  and  $\text{SO}_2$ .

**Contributions to the Knowledge of Piperidine.** C. SCHOTTEN (Vol. XV. p. 421).

Preparation of : 1. Amyl-piperidine from piperidine, amylbromide and aqueous solution of caustic potash.

2. Methyl-amyl-piperidine from amyl-piperidine and methyl iodide. Methyl-amyl-piperyl-ammonium-iodide is at first formed. The product of distillation of the ammonium base which was formed from the iodide by means of oxide of silver, almost completely dissolved in hydrochloric acid.

3. Benzyl-piperidine from benzyl-chloride and piperidine. After the reaction, water is added to the product and benzyl-piperidine is separated.

4. Methyl-benzyl-piperidine from benzyl-piperidine and methyl-iodide.

5. Nitroso-piperidine from piperidine and nitrous acid.

6. Piperyl-carbonicacidether by dropping ethyl-chloro-carbonate into piperidine. It is heavier than water and almost insoluble in it.

7. Acetyl and oxalyl-piperidine from acetyl-chloride and piperidine and oxalic ether and piperidine respectively. The author also produced pyridine from piperidine by means of bromine substitution products.

**On the Dry Products of Distillation of Tartaric Acid.** LEO LIEBERMANN. (Vol. XV. p. 428.). The products from 250 grms. of tartaric acid were:

Pyroracemic acid.....	9.24 grms.
Methylsuccinic acid.....	2.11 “
Formic acid.....	0.4 “
Tarry substances.....	3.0 “
Pitch-like substances, Aldehyde and Volatile acid.....	2.0 “

The larger proportion of the acid is hence decomposed according to this formula:



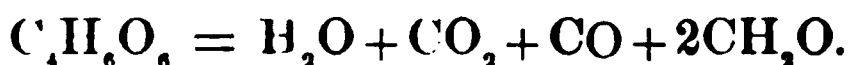
A smaller portion thus:



and a third still smaller portion:



and judging from the copious generation of carbonic acid and carbonic oxide:



**An Apparatus for Determining the Melting Point of Easily Liquified Metals and Alloys.** LEO LIEBERMANN. (Vol. XV. p. 435). Description of apparatus.

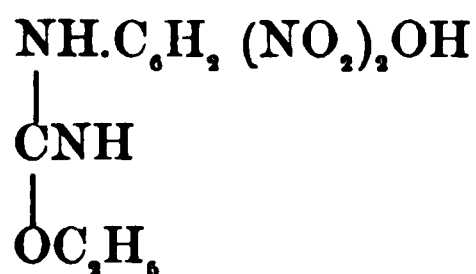
**Sulphurous Acid in Wine.** LEO LIEBERMANN. (Vol. XV. p. 437). Controversial.

**Detection of Sulphurous Acid in Wine and other Liquids.** LEO LIEBERMANN. (Vol. XV. p. 439). Two methods.

**On the Optical Power of Rotation of Malic Acid and its Salts at Different Temperatures.** Th. THOMSEN. (Vol. XV. p. 441). Results of experiments are tabulated.

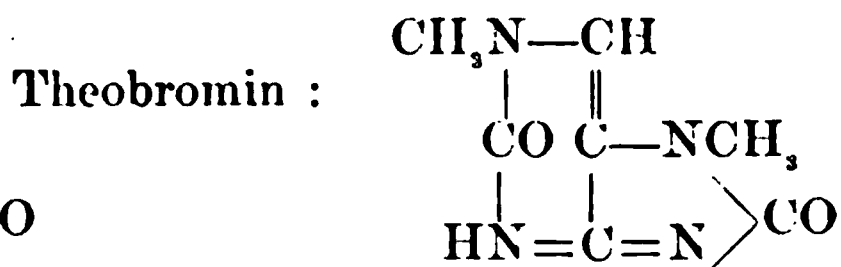
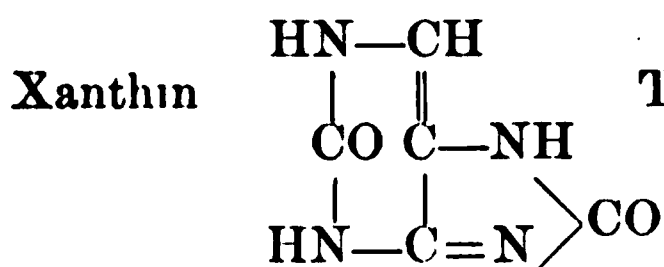
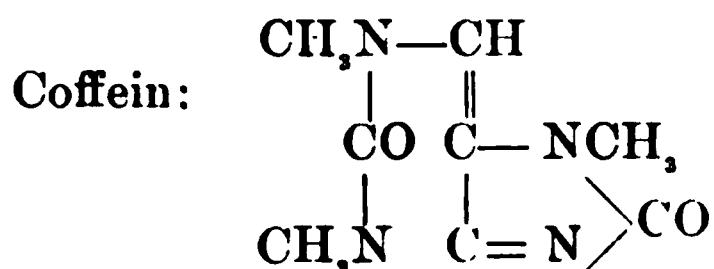
**The Action of Chlorine on Sulphonic Compounds and Organic Oxysulphides.** W. SPRING and C. WINSSINGER. (Vol. XV. p. 445). Résumé of articles on the subject published in *Bulletins de l'Académie de Belgique*; III. Série. t. II. Déc. 1881.

**On the Action of Cyanogen upon Picraminic Acid.** PETER GRIESS. (Vol. XV. p. 447). The author finds the formula for the product of a reaction between these two substances as



and terms it ethyl-oxy-carbimid-amido=dinitro-phenol.

**On the Transformation of Xanthin into Theobromin and Coffein.** EMIL FISCHER. (Vol. XV. p. 453). The author repeats an experiment, previously performed by *Strecker*, and by altering the conditions obtains evident proof of the constitution and formulæ, which latter are as follows:



**On the Action of Iodine on the Silver Salts of some Acids of the Aromatic Series.** K. BIRNBAUM and H. REINHERZ. (Vol. XV. p. 456). Experiments with benzoic, salicylic, and phthalic acids.

**On the Critical Temperatures of Liquid Bodies.** BR. PAWLEWSKI. (Vol. XV. p. 460). Controversial.

**On Tropin.** K. KRAUT. (Vol. XV. p. 462). Controversial.

**Action of Pentachloride of Phosphorous on Acetyl and Benzoyl-diphenylamin.** AL. CLAUS. (Vol. XV. p. 464). Controversial.

**On same relations between Physical Constants of Chemical Compounds** as set forth by Messrs. J. W. BRUEHL and V. ZENGER. (Vol. XV. p. 467).

**On the Action of Bisulphide of Carbon on P-nitranilin.** S. M. LOSANITSCH. (Vol. XV. p. 470). P-nitranilin and bisulphide of carbon boiled in alcoholic solution in the presence of caustic



potassa give rise to a thio-urethane, but not, as would be supposed, to a thio-urate. The analysis led to the formula



The compound is termed p-nitro-phenyl-xanthogenamid.

**On the Action of Nitric Acid on Tribromanilin (common).** S. M. LOSANITSCH. (Vol. XV. p. 471). Concentrated nitric acts energetically upon tribromanilin. The reaction is very complicated. 100 grms. of tribromanilin gave:

30 grms. dibrom-dinitro-methane.  
5 " tetrabrombenzol.  
10 " bromanil.  
24 " dark red mass.

The remaining tribromanilin is converted into picric, oxalic and carbonic acids.

The action of nitric acid on tribromanilin in glacial acetic acid solution gives rise to the same products when boiled. If allowed only to be heated until red fumes appear dibrom-nitranilin is formed.

The action of nitric acid on tribromanilin in glacial acetic acid solution and alcohol results in the production of tribrombenzol.

**On the Bases resulting from the Products of Addition of Chinalin and Halogen Alhydes.** AD. CLAUS. (Vol. XV. p. 475). Controversial.

**On the Action of Amines on Chinones.** TH. ZINCKE. (Vol. XV. p. 481). Continued from Vol. XIV.

**On the Action of Amines on Dichlor Naphtochinone.** A. PLAGEMANN. (Vol. XV. p. 484). All experiments show that only one chlorine atom can be replaced by an amine rest, and that the second chlorine atom is not capable of any such interchange. The reaction takes place in accordance with the following equation:  $\text{C}_{10}\text{H}_6\text{Cl}_2\text{O}_2 + \text{N H}_2 \text{R}' = \text{C}_{10}\text{H}_6\text{Cl}(\text{N H R}')\text{O}_2 + \text{HCl}$ . The action of primary amines proceeds smoothly, and the resulting products can be classed in general with those of alpha naphtochinan. The action of secondary amines has also been experimented upon, and they have been found to act in the same manner.

**On the Behavior of Nitrogen Tetroxide with Sulphuric Acid, and on the Process of Lasne and Benker for the Manufacture of Sulphuric Acid.** G. LUNGE. (Vol. XV. p. 488). In opposition to some previous statements, the author has

found that the tetroxide of nitrogen is not soluble as such in sulphuric acid, but is split up directly into nitrosyl sulphuric acid and nitric acid.  $N_2O_4 + SO_2(OH)_2 = SO_2(OH)(ONO) + NO_2H$ . The author proceeds to show that the theoretical assumptions of Lasne and Benker, given by these gentlemen in Vol. 92, p. 191, of *Comptes Rendus*, are not correct. This, however, does not mean that their process can not be of practical value.

**On the Existence of Nitrous Anhydride in the form of Vapor.** G. LUNGE. (Vol. XV. p. 495). Controversial.

**On the Constitution of Cumic Acid.** RICHARD MEYER and ERWIN MUELLER. (Vol. XV. p. 496). The authors prepared cumic acid synthetically, and found a different melting point from that obtained in the usual manner, but have not yet been able to give any pronounced reasons. More extensive experiments are in progress, and results may be looked for soon.

**On Tungsten Bronzes.** J. PHILLIP. (Vol. XV. p. 499). Elaborate discussion on the preparation and analysis of various bronzes. There are four distinct colors, gold yellow, reddish yellow, red and blue. The yellow bronzes are most easily prepared pure, and can be obtained in large crystals. A mixture of 2 molecules of normal sodium tungstate, and 1 molecule of tungstic anhydride is employed (hence a mixture of 2 molecules of  $Na_2O$  to 3 molecules of  $WO_3$ ). The tin is gradually added. The entire mixture is kept in quiet fusion for one or two hours. Red bronzes are obtained when 2 molecules of  $Na_2O$  and 5 or 6 molecules of  $WO_3$  are melted with tin. Blue bronzes are obtained when more than 3 molecules of  $WO_3$  to 1 molecule of  $Na_2O$  and tin are so treated. Tungsten bronzes are also prepared by reduction of acid sodium tungstates by means of hydrogen.

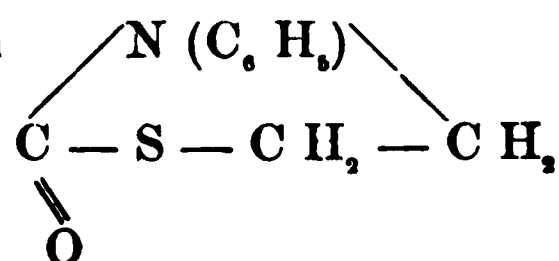
**The Azoanthrol Dyes.** C. LIEBERMANN. (Vol. XV. p. 510). The author prepared and tested a number of the dyes, and found that some of them dyed silk and wool directly, the tints produced being similar to the naphthol dyes, falling between blood red and red brown. The dyes were prepared in the usual manner, by bringing solutions of diazatised bases or their sulpho acids, in contact with alkaline solutions of anthrol.

**Contributions to the Thermometric Knowledge of Ozone.** E. MULDER and H. G. L. VAN DER MEULEN. (Vol. XV., p. 511). Berthelot found the value for  $As, O, aq 3000$  as  $137600^\circ$ , and the

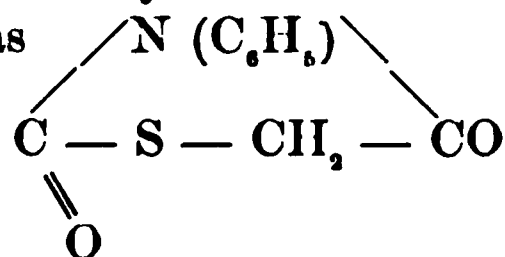
authors find that they can assume with appropriate certainty the figure 145000° for As, O, aq 2000.

**On Cyanic and Cyanuric Acid Ethers.** J. PONOMAREFF. (Vol. XV. p. 513). The object of the experimenter is to study more closely the isomerism of the neutral and acid cyanuric acid ethers which are deduced from the salts of cyanuric acid, and which are formed by the action of bromcyanogen upon sodium ethylate. Experiments are still in progress.

**On Mustard Oil Glycolide.** PAUL T. MEYER. (Vol. XV. p. 516). It having been made possible to replace oxygen in the hydro-sulpho-diphenyl-hydantoin of the group N (C<sub>6</sub>H<sub>5</sub>) and to obtain a compound having the constitution



the author definitely assumes the constitution of phenyl mustard oil glycolide as



**Action of Chlor Acetic Acid Ether on Phenylendiamin.** J. ZIMMERMANN. (Vol. XV. p. 518). The product of a reaction between monochloracetic acid ethyl ether and phenylendiamin, 2 molecules of each, is a compound to which the author gives the formula

$$\begin{array}{c} \text{CH}_2 - \text{NH} - \text{COOC}_2\text{H}_5 \\ > \text{C}_6\text{H}_4 \\ \text{CH}_2 - \text{NH} - \text{COOC}_2\text{H}_5 \end{array}$$

**Continuation of Observations on the Behavior and Occurrence of Cinchotin, Hydrocinchonidin and Hydrochinidin.** C. FORST and CHR. BOEHRINGER. (Vol. XV. p. 519). The result of experiments by the authors is that the basis obtained by oxidation were present in the original materials, and because of their great resistance remained unaltered.—*Ann. Chem. Pharm.*, 197–368.

**On the Evolution of Free Nitrogen in Fermentation.** B. E. DIETZELL. (Vol. XV., p. 551.) A series of experiments showed that nitrogen had been evolved in organic form as follows: I., 5.04 per cent.; II, 17.07 per cent.; III., 9.90 per cent.; IV., 8.97 per cent. The gases evolved from the fermentation of a mixture of blood and cow

urine were passed through dilute sulphuric acid, then over hot soda lime and titrated sulphuric acid. In all cases the sulphuric acid employed at the end remained unaltered. The author also found primary amines, leucin and free sulphurous acid. In presence of carbonate of lime solution no formation of ammonium nitrate can take place, and fatty acids which decompose nitrites cannot be formed. It seems to be advantageous to allow fertilizers, such as dried blood, bone dust, fish guano, etc., to ferment, with addition of a quantity of lime, until all nitrous acid has disappeared before they are brought into the receptacles containing the liquid excrement of domestic animals.

**On Resorcin Dyes.** G. DAMM and L. SCHREINER. (Vol. XV. p. 555.) Numerous attempts to obtain a body homologous to phenol-phatalein proved unsuccessful. A black mass was formed from which succinic acid could readily be obtained. Fusing succinic anhydride with resorcin in the air-bath at  $140^{\circ}$  resulted in the production of a body very similar to fluorescein. With bromine a body is produced which dyes similar to rosine.

**On Halogen Derivatives of Chinolins.** W. LA COSTE. (Vol. XV., p. 557.) The author prepares these derivatives in a manner differing from the original of Skraup, 86 grms. of bromanilin and the corresponding quantity of glycerin, sulphuric acid and nitrobenzol are carefully heated. The product was distilled with steam after diluting with water. The residue contained 70 grms. of pure monobromchinolin. Dinitrochinolin and phenylchinolin were prepared by similar methods.

**On Normal Dithiurethanes.** A. BERNTHSEN and G. FRIESE. (Vol. XV., p. 563.) Partly controversial. The results of experiments agree satisfactorily with those of W. Will. Ber. d. d. chem. Ges. XV. 338.

**Remarks on a Note of C. Binzard and H. Schulz: On the Chemical Theory of Effects of Arsenic** J. DOGIEL. (Vol. XV., p. 572.) Controversial.

**Remarks on M. Foerster's Article on the Presence of Furfurol in Fermented Liquids.** W. A. JORISSEN. (Vol. XV., p. 572.) Controversial.

**The Condensation of Acetone.** A. PINNER. (Vol. XV., p. 576.) Continued from Vol. XIV., p. 576. Cyanide of potash was to act upon acetone which allowed had been saturated with hydro-

chloric acid gas and thus condensed. Three combinations which could be easily separated were obtained; an acid  $C_7H_{12}O_3$  called mesitonic acid, an acid of the composition  $C_{11}H_{13}NO_3 + H_2O$  called (for the present) mesitylic acid, and a cyanide  $C_{11}H_{11}N_2O_3$  from which an acid  $C_{11}H_{13}O_3$  (phoronic acid) could be obtained.

**Formation of Alloys by Pressure.** W. SPRING. (Vol. XV., p. 595.) The author propounds the law, that the state of matter is dependent upon the volume it is forced to occupy. Coarsely powdered bismuth, cadmium and tin, mixed in the proportions corresponding to Wood's alloy, were subjected to a pressure of 7,500 atmospheres. This alloy melts at  $65^\circ$ . The block obtained was filed and again subjected to the same pressure. Its properties correspond exactly with those of Wood's alloy. Two other alloys, one known as Rose's and common brass, were treated in a similar manner, and the results obtained gave sufficient strength for the argument of the law set forth.

**On Orthodinitro Compounds.** AUG. LAUBENHEIMER. (Vol. XV., p. 597.) Continued from Vols. IX., 768 and 1826 ; XI., 1155; XI., 303; IX., 760. The author finds that orthodinitrocompounds also react with sulphite of sodium.

**On Bromchloral, Chlorobromal, Bromochloroform and Chlorobromoform.** OSCAR JACOBSEN and R. NEUMEISTER. (Vol. XV., p. 599.) Description of these compounds obtained by the action of bromine on dichloroacetal and monochloroacetal and further decomposition of the products produced.

**Bromdichloroacetic Acid and Chlordibromoacetic Acid.** R. NEUMEISTER. (Vol. XV., p. 602.) When bromochloral or chlorobromal is heated with double its quantity of fuming nitric acid for about one hour, with vertical condenser, the corresponding substituted acetic acid crystallizes from it after cooling. Bromdichloroacetic acid is  $CCl_2Br.CO_2H$  and chlordibromoacetic acid is  $CClBr_2.CO_2H$ . A number of derivatives of both compounds are described.

**Note on Bodies Combustible with Difficulty.** W. DEML (Vol. XV. p. 604.)

In elementary analysis some substances are burned incompletely by the usual methods. The author finds an improvement in covering the substance to be analyzed in the platinum boat with three or four times its weight of previously ignited spongy platinum or platinum black.

**On Chloromalonic Acid and its Derivatives.** M. CONRAD and M. GUTHZEIT. Vol. XV. p. 605.

The free acid was produced from monochloromalonic acid ether by means of the potash salt. When monochloromalonic acid is heated until all carbon acid is driven off monochloroacetic acid remains. By the action of ammonia upon monochloromalonic acid imido-dimalonyl-amid and amido-malonyl-amid were produced.

**On Propylic and Isopropyl Succinic Acid.** G. WALTZ. (Vol. XV. p. 608.)

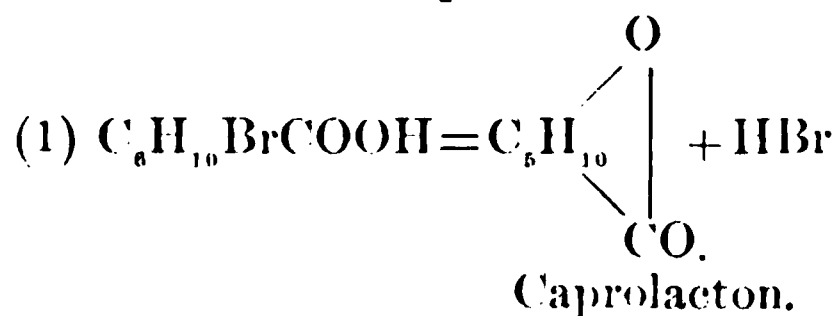
Description of method of preparation of derivatives.

**On the Transformation of Alpha and Betanaphtal into Amidated Naphtalines.** ARTHUR CALM. (Vol. XV. p. 609.)

Beta naphtylamine was obtained by the action of acetamid (ammoniumacetate) after distillation. The residue was found to be in part beta-acet-naphtalide, and in part beta-dinaphtylamine. A table giving the various temperatures and time required and results is appended.

**On Caprolactone.** ED. HJELT. (Vol. XV. p. 617.)

The caprolactone was obtained from monobrom-capranic acid with water. The reactions take place as follows:



**Action of Water on Isodibromcapronic Acid.** ED. HJELT. (Vol. XV. p. 619.)

A mixture of a lactone containing bromine and of a lactone free from bromine were the result of boiling dibromcapronic acid for one hour with ten times its weight of water. All bromocapronic acids prepared from sorbinic and hydrosorbinic acids, when heated with water or alkaline solution produce lactones.

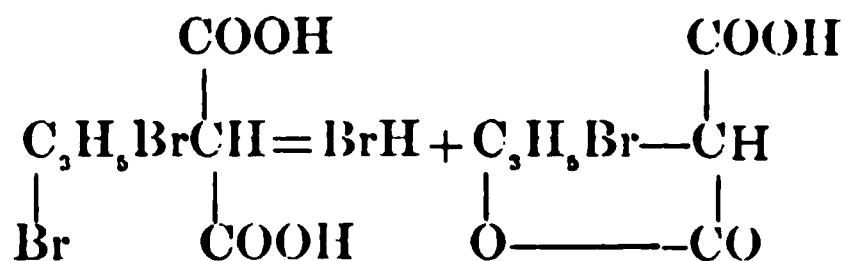
**Oxypropylmalonic Acid and its Lactone.** ED. HJELT. (Vol. XV. p. 621.)

Allylmalonic acid dissolves quite readily in fuming hydrobromic acid. The acid formed was found to be the lactonic acid, having

the same composition as the allylmalonic acid. It is a monobasic lactonic acid, the lactone of an oxypropylmalonic acid.

**Action of Bromine on Allylmalonic Acid.** ED. HJELT. (Vol. XV. p. 624.)

Besides the dibromide an oil poorer in bromine is produced. The reaction takes place as follows:



**On Dilactones.** ED. HJELT. (Vol. XV. p. 625.)

By the action of 2 mol. of bromine upon diallylmalonic acid a dibromide is produced. Hydrobromic acid is freely produced. The quantity of bromine present as hydrobromic acid after the reaction is completed was 50 per cent. of the amount of bromine employed. The bromide being a neutral body its formation can only be comprehended by assuming the formation of a tetrabromide at first, from which 2 mol. of bromine were driven which combined with the two hydrogen atoms of the carboxyls. The compound is hence an example of a dilactone.

**Action of Hydrobromic Acid and Bromine on Diallylacetie Acid.** ED. HJELT. (Vol. XV. p. 627.)

Hydrobromic acid reacts with diallylacetie acid forming a thick oil termed brom-dipropyl-aceto-lactone. Bromine reacts with it, producing tribrom-dipropyl-aceto-lactone.

**On the Boiling Points of Lactones.** ED. HJELT. (Vol. XV. p. 629.)

Gives the boiling points of lactones hitherto discovered.

**On the Formation of Lactone.** ED. HJELT. (Vol. XV. p. 629.)

From the researches of Fittig it is comparatively certain that the lactones are generated from the oxyacids and bromine substitution acids, which have their hydroxyl group or the bromine atom respectively attached to the third carbon atom, beginning at the carboxyl group. For this theory the investigations of the author on the addition products of bromine and hydrobromic acid with allylmalonic, diallylmalonic and diallylacetie acids give sufficient evidence.

Abstracts from the Comptes Rendus; by A. Bourgougnon.

**Action of Ethylenic Chlorhydrine upon the Pyridic bases and Quinolein.** By A WURTZ. (No. 6, Aug. 7th, 1882).

**Upon the Composition of the Iodides of Phosphorus.** By L. FROOST. (No. 6. Aug. 7th, 1882).

The composition of the Iodides of Phosphorus is represented by  $PI_3$  and  $P_2I_4$ .

**Upon Quinolein and Lutidin.** By A. PICTET. (No. 6. Aug. 7th, 1882).

**Neutral Arsenates.** By E. FILHOL & SENDERN. (No. 7. Aug. 14th, 1882).

The arsenates  $2 As_2O_3, 3 Na. OH + 6 H_2O$  and  $+ 4 As_2O_3, 3 Na OH + 3 KOH + 18 H_2O$  have no action upon litmus.

**Upon the Chemical Composition of the ripe and unripe Banana.** By L. RICCIARDI. (No. 8. Aug. 21st, 1882).

	Ripe.	Unripe.
Water.....	66.78	7.92
Cellulose.....	0.17	0.36
Starch.....	traces	12.06
Tannic substances.....	0.34	6.53
Fatty ".....	0.58	0.21
Interverted sugar.....	20.07	0.08
Cane sugar.....	4.50	1.34
Proteic substances.....	4.92	3.04
Not determined.....	1.69	4.42
Ashes.....	0.95	1.04

The green banana contains a large amount of starch, about  $\frac{1}{2}$  of its weight which disappears in the ripe fruit. When the fruits are left to ripen on the trees nearly all the sugar formed is cane sugar, whilst the sugar contained in the fruits taken from the trees in a green state and allowed to ripen afterwards is composed of  $\frac{4}{5}$  of intervverted sugar and  $\frac{1}{5}$  of cane sugar. The tannic substances and the organic acids of the green fruits disappears when the bananas are ripe.

The ashes of bananas are very rich in phosphoric acid (23.18 per cent.) and potassium oxide (45.23 per cent.)

**On Black Phosphorus.** By P. THENARD. (No. 9. Aug. 28th, 1882).

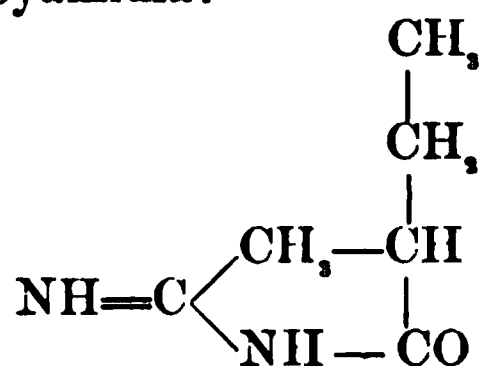


**Separation of Gallium.** By L. DE BOISBAUDRAN. (No. 9. Aug. 28th, 1882).

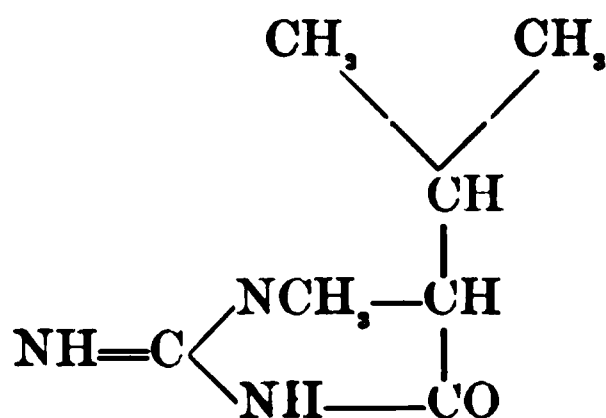
Separation of Gallium from Indium and Cadmium.

**On some combinations belonging to creatin groups.** By E. DUVILLIER. (No. 10, Sept. 4th, 1882).

The following combinations have been obtained : 1° Methylamido Alpha-butyrocyamidin:



2° Methylamids isovalerocyamidin.



**Separation of Gallium.** By L. de BOISBAUDRAN. (No. 12 Sept. 18th, 1882).

Separation of Gallium from Uranium and Lead.



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P. DEP. RICKETTS.

# HONORARY MEMBERS.

---

BERTHELOT, M., COLLEGE OF FRANCE, PARIS.'

BOUTLEROW, A., UNIVERSITY OF ST. PETERSBURG, ST. PETERSBURG,  
RUSSIA.

BUNSEN, R. W., UNIVERSITY OF HEIDELBERG, GERMANY.

CANNIZZARO, STANISLAS, UNIVERSITA ISTITUTO CHEMICO DELLA  
REGIA, ROME, ITALY.

FRANKLAND, E., ROYAL COLLEGE OF CHEMISTRY, SOUTH KENSINGTON  
MUSEUM, LONDON, ENGLAND.

WILLIAMSON, A. W., UNIVERSITY COLLEGE, LONDON, W. C., ENGLAND.

WOEHLER, FR.\*

## MEMBERS.

LIFE MEMBERS ARE MARKED |.

DECEASED MEMBERS ARE MARKED \*.

### DATE OF ELECTION.

April	6, 1876	Adams, Isaac, Jr., 329 Broadway N. Y.
Dec.	7, 1876	Aitken, Andrew P., Clyde S., Edinburgh, Scotland.
April	6, 1876	Alsberg, M., 54 Maiden Lane, N. Y.
April	6, 1876	Amend, B. G., 205 Third ave., N. Y.
May	8, 1877	Appleton, J. H., Brown University, Providence, R. I.
April	6, 1876	Atwater, Wilbur O., Middletown, Conn.
Dec.	7, 1876	Austen, P. T., Rutger's College, New Brunswick N. J.
July	6, 1876	Bailey, E. H., Bethlehem, Pa.
March	7, 1878	Baker, W. H., Freeport, Ill.
April	6, 1876	Balentine, Walter, Argicultural Department, Washington, D. C.
Oct.	5, 1876	Barcena, Mariano, Museo Nacional, City of Mexico, Mexico.
April	6, 1876	Battershall, J. P., 76 Varick st., N. Y.
Oct.	7, 1881	Bauer, Harry von, 12 Frankford st., N. Y.
June	1, 1876	Baumgarten, A., St. Lawrence Sugar Refining Co., Montreal, Canada.
April	6, 1876	Baumgarten, F. M., 179 16th st., San Francisco, Cal.
Jan.	3, 1878	Beckwith, Leonard F., Courtlandt and Church sts., N. Y.
April	6, 1876	Behr, Arno, care of Matthiessen & Wiechers, Jersey City N. J.
July	6, 1876	Belcher, G. W., St. Louis, Mo.
April	3, 1879	Benjamin, Marcus, 6 Barclay st., N. Y.
Oct.	7, 1880	Bihn, G. T., 523 Tasker st., Philadelphia, Pa.
Nov.	1, 1877	Blanpied, Benj., Dartmouth Agricultural Coll., Hanover, N. H.
April	6, 1876	*Blossom, Thomas M.
Dec.	7, 1876	Booth, J. C., United States Mint, Philadelphia, Pa.
April	6, 1876	Bourgougnon, A., 26 W. 4th St., N. Y.
March	1, 1880	Bragg, Everett B., 33 Park place, N. Y.
Jan.	6, 1882	Braschi, Victor M., School of Mines, N. Y.
April	7, 1882	Brenneman, A. A., Cornell Univ'y, Ithaca N. Y.
April	6, 1876	Brinley, Charles A., Midvale Steel Works, Germantown, Phila- delphia, Pa.
Dec.	6, 1877	Brown, Wm. G., East Tennessee University, Knoxville, Tenn., Box 596.
Sept.	7, 1876	Buck, C. Elton, Wilmington, Del.

## DATE OF ELECTION.

April	6, 1876	*Cairns, F. A.,
May	2, 1878	Calder, E. E., Box 17, Providence, R. I.
April	6, 1876	Casamajor, P., care Havemeyer & Elder, Brooklyn, E. D., N. Y.
April	6, 1876	Chandler, Chas. F., School of Mines, Columbia College, N. Y.
Sept.	7, 1877	Chase, Alonzo, Room 123, Cochrane B'ld'g, Washington, D. C.
April	6, 1876	Chandler, W. H., Bethlehem, Pa.
April	6, 1876	Chester, Albert H., Clinton, N. Y.
Oct.	7, 1881	Colby, Albert L., School of Mines, N. Y.
March	7, 1878	Colby, E. Edwards, School of Mines, Columbia College N. Y.
Dec.	6, 1879	Convers, G. G., U. S. Mint. New Orleans, La.,
Feb.	1, 1877	Corning, A. J., N. E. cor. Bolton & Mosher sts., Baltimore, Md
Nov.	7, 1878	Cruse, E. F., 20 Bridge st., Brooklyn, N. Y.
Dec.	6, 1877	Dabney, Charles W., Raleigh, N. C.
Nov.	4, 1881	Darton, Nelson H., 119 Pearl st., N. Y.
Sept.	5, 1878	Debrunner, Henry G., Iron City, via Alpine Chaffee Co., Co.
April	6, 1876	Degenhardt, F. A. C., 333 S. 5th st., Brooklyn, N. Y.
April	6, 1876	Doremus, Charles A., 92 Lexington ave. N. Y.
Dec.	2, 1879	Dosher, Henry, 142 Rose st., Brooklyn, E. D.
April	6, 1876	Douglass, Silas H., Ann Arbor, Mich.
April	6, 1876	* Draper, John W.
Nov.	2, 1876	Dudley, Charles G., Altoona, Pa
July	6, 1876	Dunnington, F. P., University of Virginia, Albemarle Co., Va.
May	3, 1877	Dwight, Charles Edgar, Wheeling, W. Va.
April	6, 1876	Eastwick, E. P., 78 Wall st., N. Y.
April	6, 1876	Ebert, A. E., Peoria Sugar Refinery, Peoria, Ill.
March	6, 1879	Eimer, August, 220 E. 17th st., N. Y.
April	6, 1876	Elliott, Arthur H., School of Mines, Columbia College, N. Y.
Oct.	5, 1876	Ellis, William H., Toronto, Canada.
April	6, 1876	Endemann, H., 33 Nassau st., N. Y.
April	6, 1876	Englehardt, Francis E., Syracuse Salt Co., Syracuse, N. Y.
March	6, 1879	Erhardt, C. F., care Chas. Pfizer & Co., 81 Maiden Lane N. Y.
April	6, 1876	Fesquet, Adolphe A., 1322 Marshall st., Philadelphia, Pa.
June	1, 1876	Fisher, R. A., 2239 St. Albans st., Philadelphia, Pa.
Dec.	2, 1879	Freish, H., 136 Church st., Bridenburg, Philadelphia, Pa.
Dec.	2, 1880	Friedburg, L. H., 40 E. 10 st., N. Y.
April	6, 1876	Fristoe, Edward E., Columbian College, Washington, D. C.
May	1, 1879	Gallatin, Albert H., Room 26, Cooper Union, N. Y.
April	6, 1876	Genth, Fred. A., University of Pennsylvania, Phila., Pa.
Feb.	11, 1881	Gerber, N., Amer. Cond. Milk Co., Little Falls, N. Y.
April	6, 1876	Gesner, G. W., 81 John st. N. Y.
March	1, 1877	Geyer, Wm. E., Stevens Institute, Hoboken, N. J.
Oct.	14, 1879	Giessecke, Dr., 19 W. Swan st., Buffalo, N. Y.
April	2, 1882	Gladding, Thomas S., 55 Fulton st., N. Y.
April	6, 1876	Goessman, Charles A., Amherst, Mass.
April	6, 1876	* Goldmark, J.
Oct.	5, 1876	Goldschmidt, S. A., 55 Water St., Brooklyn.

## DATE OF ELECTION.

April	6, 1876	Green, Trail, Easton, Pa.
June	7, 1877	Gregg, W. H., Elmira, N. Y.
May	6, 1881	Grote, Otto, 59-61 Goerck st., Y. Y.
April	6, 1876	Grund, F. A., 106 Wall st., N. Y.
April	6, 1876	Habirshaw, William M., 159 Front st., N. Y.
Dec.	2, 1880	Hale, Albert C., State School of Mines, Golden, Col.
June	8, 1881	Hallock, A. P., N. Y. Gaslight Co., ave. A and 21st st., N. Y.
Oct.	7, 1881	Habel, Louis, 287 Leonard st., Brooklyn, N. Y.
Oct.	2, 1879	Hamer, Thomas H., Nassau Bank, N. Y.
April	6, 1876	Hardin, M. B., Virginia Military Institute, Lexington, Va.
Oct.	7, 1880	Harrison, Thomas, Philadelphia, Pa.
April	6, 1876	Hasenclever, Maurice, 1 Appleton st., Lawrence. Mass.
June	1, 1876	Havemeyer, Hector C., 89 Wall st., N. Y.
April	6, 1876	Hayes, S. Dana, 4 State st., Boston, Mass.
Feb.	6, 1879	Hazard C. care Caswell, Hazard & Co., 5th Ave. Hotel, N. Y.
April	6, 1876	Hedrick, Benjamin S., U. S. Patent Office, Washington, D. C.
June	1, 1876	Henes, W. F., 1620 Third ave., N. Y.
April	6, 1876	Herreshoff, J. B. F., 135 Clinton st., Brooklyn, N. Y.
Oct.	7, 1881	Hiepe, H. C., 102 John st., N. Y.
April	6, 1876	Hill, Nathaniel P., Denver, Col.
Jan.	4, 1877	Hill, W. M., U. S. Torpedo Station, Newport, R. I.
March	1, 1877	Hindley, Robert C., Racine College, Racine, Wis.
March	8, 1882	Hirshberg, Emil, 16 Dey st., N. Y.
April	6, 1876	Homer, Chas. S., care of Valentine & Co., 323 Pearl st., N. Y.
Feb.	11, 1881	Hopke, T. M., 17 Cedar st., N. Y.
April	1, 1881	Hoppock, A. E., 103 E. 148th st., N. Y.
Jan.	4, 1877	Horsford, E. N., Cambridge, Mass.
April	6, 1876	Humphrey, Henry C., Stamford, Ct.
April	6, 1876	Hunt, T. Sterry, 33 Durocher st., Montreal, Canada.
June	6, 1878	Jenks, A. M., care of Havemeyer, Eastwick & Co., Jersey City, N. J.
July	6, 1876	Johnson, Samuel W., New Haven, Ct.
March	6, 1879	Kelly, John F., 97 Garden st., Hoboken, N. J.
April	6, 1876	Kersting, Herman, 324 Princeton st., East Boston, Mass.
July	6, 1876	Koenig, G. A., University of Pennsylvania, Philadelphia, Pa.
April	6, 1876	Krackowitzer, S., 512 E. 17th st., N. Y.
April	6, 1876	Krause, O. H., care Matthiessen & Wiechers, Jersey City, N. J.
April	7, 1882	Krause, G. Chemiker Zietung, Coethen, Germany.
Oct.	7, 1881	Lauver, Winfield P., care of Treasury Department, Washington, D. C.
July	6, 1876	Lawler, C. J., cor. Bedford and Gates aves., Brooklyn, N. Y.
Dec.	7, 1876	Lecour, E. H., 547 W. 18th st., N. Y.
April	6, 1876	Ledoux, A. R., 17 Cedar st., N. Y.
Jan.	3, 1878	Leeds, A. R., Stevens Institute, Hoboken, N. J.
Jan.	3, 1878	Lees, Wm. T., N. Y. Gaslight Co., ave. A and 31st st., N. Y.
April	6, 1876	Liebig, G. A., 87 Exchange place, Baltimore, Md.



## DATE OF ELECTION.

July	6, 1876	Lillie, Samuel Morris, 883 Pine st., Philadelphia, Pa.
Oct.	7, 1881	Leman, W. P., 305 W. 55th st., N. Y.
April	6, 1876	Lindsley, J. Berrian, Nashville, Tenn.
April	6, 1876	*Lipps, John S.
Feb.	8, 1882	Lugo, Orazio, University, Burling, N. Y.
Dec.	2, 1880	Lungwitz, Theo., care of H. E. Niese, Jersey City, N. J.
April	6, 1876	Lupton, N. T., Vanderbilt University, Nashville, Tenn.
May	2, 1878	Luthy, D., 220 Church st., Philadelphia, Pa.
April	6, 1876	McIntire, Chas. M., Jr., 311 N. Hampton st., Easton, Pa.
Jan.	2, 1879	*McIntire, Henry M.
June	6, 1878	MacIntosh, J. B., 55 Garden st., Hoboken, N. J.
April	6, 1876	Maish, John M., College of Pharmacy, Philadelphia, Pa.
April	6, 1876	Mallet, J. W., University of Virginia, Albemarle Co., Va.
Feb.	6, 1879	Marchand, Charles, P. O. Box 1567, N. Y.
April	6, 1876	Martin, Wm. J., Davidson College, Mecklenburg Co., N. C.
Sept.	7, 1876	Martins, R. A., care of Actien-Gesellschaft fuer Anilin Fabrika. tion, Berlin, Germany.
May	6, 1878	Mattison, J. G., Hastings-on-Hudson, N. Y.
Feb.	1, 1877	Means, Alexander, Oxford, Newton Co., Ga.
April	6, 1876	*Merrick, John M.
April	6, 1876	Mew, Wm. M., 422 5th st., Washington, D. C.
Dec.	1, 1879	Meyers, Henry C., P. O. Box 3037 N. Y.
June	1, 1876	Miller, George M., 20 Broad st., N. Y.
April	6, 1876	Moore, Gideon E., 69 Liberty st., N. Y.
April	6, 1876	Morrill, Thomas T., Cambria Iron Works, Johnstown, Pa.
April	6, 1876	Morton, Henry, Stevens Institute, Hoboken, N. J.
June	1, 1871	Mott, H. A. Jr., 105 Water st., N. Y.
April	6, 1876	Munroe, Charles E., U. S. Naval Academy, Annapolis, Md.
March	7, 1878	Munroe, H. S., School of Mines, Columbia College, N. Y.
Nov.	4, 1881	Munsell, C. E., 128 Worth st., N. Y.
April	6, 1876	Nason, Henry B., Troy, N. Y.
April	6, 1876	Niese, H. E., care of Matthiessen & Wiechers, Jersey City, N. J.
April	6, 1876	Nichols, W. H., 41 Cedar st., N. Y.
May	3, 1877	Norton, Thomas, 2 Route Landai, Saint Denis, Paris, France.
Nov	4, 1881	O'Connor, Thos. D., 42 W. 28th st., N. Y.
Dec.	2, 1880	O'Connor, J. D., School of Mines, Columbia College, N. Y.
Nov.	19, 1879	Oothout, William, Jr., 218 Madison ave. N. Y.
Dec.	6, 1877	Orth, A., care of F. Bredt & Co., N. Y.
July	6, 1876	Parker, T. J., Box 69, Bergen Point, N. J.
April	6, 1876	Parsons, C. C., 542 E. 20th st., N. Y.
July	6, 1876	Pearce, Richard, Denver, Col.
May	3, 1877	Penissat, A., 41 Cedar st., N. Y.
April	7, 1882	Perkins, G. E., Providence, R. I.
April	6, 1876	Perkins, Maurice, Schenectady, N. Y.

## DATE OF ELECTION.

Jan.	6, 1882	Peters, J. A., 92 William st., N. Y.
April	6, 1876	Phelps, George N., 127 First place, Brooklyn, N. Y.
July	11, 1878	Phillips, William B., University of North Carolina, Chapel Hill, N. C.
Nov.	2, 1876	Pickhardt, Carl W., Pickhardt & Kuttroff, cor. of Liberty and New Church sts., N. Y.
Dec.	2, 1880	Pigeon, Narcise, 421 W. 15th st., N. Y.
April	6, 1876	Pirz, Anthony, Long Island City, N. Y.
Oct.	5, 1876	Pitkin, Charles A., Box 52, Braintree, Mass.
Jan.	6, 1882	Pitkin, Lucius, 432 Madison ave., N. Y.
March	6, 1879	Pool, Franklin V., 191 Roseville ave., Newark, N. J.
Oct.	7, 1880	Poole, Herman, Buffalo, N. Y.
April	6, 1876	Prescott, Albert B., Ann Arbor, Mich.
March	1, 1877	Prochazka, George A., 162 2d ave., N. Y.
Oct.	7, 1881	Radenhausen, P., Stevens Institute, Hoboken, N. J.
Jan.	6, 1882	Rau, Henry, 92 Church st., N. Y.
April	7, 1882	Remsen, Robert G., Jr., 26 Waverly place, N. Y.
April	6, 1876	Rice, Charles, Bellevue Hospital, N. Y.
April	6, 1876	Ricketts, P. de P., School of Mines, Columbia College, N. Y.
April	6, 1876	Rising, William B., University of California, Oakland, Cal.
June	1, 1876	Robbins, Charles A., 91 Fulton st., N. Y.
March	4, 1878	Roberts, W. Chandler, Royal Mint, London, Eng.
March	4, 1878	Rogers, F. M., 95 Liberty st., N. Y.
April	6, 1876	Rogers, Robert E., Jefferson Medical College, Phila., Pa.
Nov.	1, 1877	Rossi, Auguste J., 22 W. 11th st., N. Y.
July	11, 1878	Rupp, William, 117 Pearl st., N. Y.
April	6, 1876.	*St. John, Samuel.
June	5, 1879	Schanck, J. Stillwell, Princeton College, Princeton, N. J.
April	6, 1876	Schultz, Charles H., cor. 17th st. and Broadway, N. Y.
April	6, 1876	Schweitzer, Paul, Columbia, Boone Co., Mo.
Dec.	2, 1880	Seaman, Henry J., Crane Iron Co., Catasauqua, Pa.
July	6, 1876	Seeley, Henry Martyn, Middlebury, Vt.
Jan.	1, 1880	Semper, C., Gray's Ferry Chem. Works, Philadelphia, Pa.
April	6, 1876	Shapleigh, Waldron, Freeport, Ill.
Nov.	4, 1881	Share, W. W., 336 Navy st., Brooklyn, N. Y.
April	6, 1876	Sharpless, S. P., 114 State st., Boston, Mass.
Oct.	3, 1878	Shepard, Charles U., jr., 20 Broad st., Charleston, S. C.
April	6, 1876	Sherer, E., 122 Front st., N. Y.
April	6, 1876	Silliman, Benjamin, New Haven, Ct.
April	6, 1876	Simon, Wm., Maryland College of Pharmacy, Baltimore, Md.
Feb.	11, 1881	Slade, James F., 5 E. 38th st., N. Y.
May	3, 1877	Sloane, T. O'C., 55 Pine st., N. Y.
Nov.	1, 1877	Smith, Hanbury, 35 Union square, N. Y.
March	1, 1877	Squibb, E. R., 36 Doughty st., Brooklyn, N. Y.
June	5, 1879	Stebbins, James H., Jr., 20 E. 37th st., N. Y.
April	6, 1876	Sticht, John C., care of Charles Pfizer & Co., 81 Maiden Lane, N. Y.

## DATE OF ELECTION.

April	6, 1876	Stillwell, Charles M., 55 Fulton st., N. Y.
July	6, 1876	Sweeny, William S., Easton, Pa.
April	6, 1876	Taylor, William H., 606 E. Grace st., Richmond, Va.
Oct.	5, 1876	Terne, Bruno, 1628 S. 5th st., Philadelphia, Pa.
Feb.	7, 1878	Thompson, A. L., 52 Sharp st., Baltimore, Md.
May	1, 1881	Touneîé, Theodore, Wampum Cement and Lime Co., New-castle, Lawrence Co., Pa.
Oct.	7, 1880	Torrey, C. H., School of Mines, N. Y.
Feb.	7, 1878	Tscherniac, J., 118 Boulevard Maillot, Neuilly, Paris, France.
May	6, 1879	Tucker, J. H., care of McKean, Newhall & Borie, Philadel-phia, Pa.
April	3, 1879	Van Slooten William, 42 S. Derbigny st., New Orleans, La.
April	6, 1876	Vaughn, Henry W., Providence, R. I.
Feb.	11, 1881	Venable, F. P., Chapel Hill, N. C.
Nov.	4, 1881	Volney, C. W., Mott ave., N. Y.
Dec.	6, 1877	Wait, Charles E., Missouri School of Mines, Rolla, Mo.
Jan.	3, 1878	Waldstein, Martin E., 92 New Church st., N. Y.
April	6, 1876	Waller, Elwyn, School of Mines, Columbia College, N. Y.
April	6, 1876	*Walz, Isidor.
June	6, 1878	Ware, Louis S., St. George Hotel, Philadelphia, Pa.
April	6, 1876	Weber, Henry A., Ill. Industrial University, Champaigne, Ill.
April	6, 1876	Wendt, Charles J., care of Charles Pfizer & Co., 81 Maiden Lane, N. Y.
Sept.	7, 1876	Wenzell, Wm. T., 852 Market st., San Francisco, Cal.
March	6, 1879	Wheeler, C. G., University of Chicago, Chicago, Ill.
Dec.	2, 1880	Wiechman, F. G., School of Mines, N. Y.
Jan.	2, 1879	Wigner, G. W., 79 Great Tower st., London, England.
Dec.	1, 1879	Williams, E. H., 101 N. 32d st., Philadelphia, Pa.
June	5, 1879	Wilson, M. C., Waco, Texas.
April	6, 1876	Winans, Horatio N., 1551 Broadway, N. Y.
Nov.	4, 1881	Witthaus, R. A., Buffalo Med. College, Buffalo, N. Y.
Dec.	2, 1880	Wittman, Byron N., Pittsburg Steel Casting Co., Pittsburg, Pa.
July	6, 1876	Wolf, Theo. R., Delaware College, Newark, Del.
April	6, 1876	Wormley, Theo. G., University of Pennsylvania, Phila., Pa.
July	6, 1876	Ycumans, W. J., care of D. Appleton & Co., 1 Bond st., N. Y.

## ASSOCIATE MEMBERS.

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**DATE OF ELECTION.**

June	1, 1876	Adler, J., 744 Lexington ave., N. Y.
June	1, 1876	Bartlett, Willard, 110 Broadway, N. Y.
March	6, 1879	Binns, Joseph, cor Pearl and Cedar sts., N. Y.
March	4, 1880	Booraem, J. V. V., N 8d and 1st sts., Brooklyn, E.D., N. Y.
June	6, 1878	Bower, Henry, Gray's Ferry Road, Philadelphia Pa.
June	3, 1880	Cochrane, A., 55 Kilby st., Boston, Mass.
June	3, 1880	Cochrane, Hugh, 55 Kilby st., Boston, Mass
July	6, 1876	Day, Austin G., 120 Broadway, N. Y.
June	1, 1876	Day, Walter De F., 301 Mott st., N. Y.
July	6, 1876	Dickerson, Edward N., Staats-Zeitung Building, N. Y.
April	5, 1877	Donner, J. O., care of Decastro & Donner, foot of South 9th st., Brooklyn, N. Y.
Feb.	11, 1881	Dunham, E. K., School of Mines, N. Y.
Jan.	4, 1877	Elder, Fred H., care of Havemeyer & Elder, 117 Wall st., N. Y.
July	6, 1876	Elder, George. Jr., 19 West 48th st., N. Y.
Oct.	5, 1876	Elmenhorst, W. R., St, Lawrence Sugar Refining Co., Montreal, Canada.
July	6, 1876	Gandolfo, Joseph, 41 Cedar st., N. Y.
April	4, 1878	Harding, G., 237 Broadway, N. Y.
Sept.	7, 1876	Havemeyer, C. W., care of Havemeyer Bros., Brooklyn, E. D., N. Y.
April	5, 1877	Havemeyer, Henry, 89 Wall st., N. Y.
July	6, 1876	Havemeyer, Henry O., 117 Wall st., N. Y.
July	6, 1876	Havemeyer, Theodore A., 117 Wall st., N. Y.
April	5, 1877	Heller, Fred., 55 Maiden Lane, N. Y.
June	1, 1866	Hinds, Joseph, care of Havemeyer & Elder, Brookly, E. D., N. Y.
Dec.	6, 1876	Holly, John J., 63 Beaver st., N. Y.
June	1, 1876	Holt, Henry, 12 East 23d st., N. Y.
April	5, 1877	Kalbfleisch, A. M., cor. Fulton and Cliff sts., N. Y.
April	5, 1877	Kalbfleisch, Franklin A., cor. Fulton and Cliff sts., N. Y.
Nov.	7, 1876	Kuttroff, Adolph, Wm., Pickhardt & Kuttroff, cor. New Church and Liberty sts., N. Y.
Dec.	2, 1880	Kyte, Charles, Jr., 3 East India ave., London, E. C.

## DATE OF ELECTION.

Jan.	6, 1882	Le Boutillier, Clement, School of Mines, N. Y.
Feb.	6, 1879	Leggett, E. W., Elizabethport, N. J.
March	1, 1877	Lennig, Charles, Philadelphia. Pa.
July	6, 1876	Mapes, Charles V., 158 Front st., N. Y.
July	6, 1876	Matthiessen, F. O., 106 Wall st., N. Y.
April	5, 1877	Merz, Henry, care of Heller & Merz, 55 Maiden Lane, N. Y.
July	6, 1877	Miles, William A., 57 Christie st., N. Y.
April	5, 1877	Nichols, G. H., 41 Cedar st., N. Y.
Sept.	7, 1876	Page, George, Shepherd, 49 Wall st., N. Y.
July	6, 1876	Parsons, W. J., 66 Water st., Brooklyn, N. Y.
July	6, 1876	Plympton, G. M., 245 Broadway, N. Y.
Nov.	2, 1876	Pickhardt, William, Wm. Pickhardt & Kuttroff, cor. New Church and Liberty sts., N. Y.
March	7, 1878	Prentice, W. P., 801 Mott st., N. Y.
May	3, 1877	Renwick, Edward S., 16 Murray st., N. Y.
Sept.	7, 1876	*Russell, W. D.
Jan.	6, 1882	Rudesch, J., 117 East 59th st.
May	6, 1881	Sawyer, C. P., High Point, Guilford Co., N. C.
Oct.	5, 1876	Senff, Charles H., care of Havemeyer & Elder, 117 Wall st., N. Y.
July	6, 1876	Senff, Frederick W., 558 Madison ave., N. Y.
April	5, 1877	Schroeder, E., 100 Glenwood ave., Jersey City, N. J.
April	5, 1877	Siegheortner, A. L., Belcher Sugar Refining Co., St. Louis, Mo.
Jan.	4, 1877	Squier, Stewart C., 118 Liberty st., N. Y.
April	5, 1877	Stursberg, Julius A., Brooklyn Sugar Refinery, foot of South 9th st., Brooklyn, N. Y.
May	6, 1881	Van Sinderen, A. Howard, School of Mines, N. Y.
Dec.	2, 1880	Vulte, Herman T., School of Mines, N. Y.
May	2, 1878	Weston, E. D., 135 Lake st., Chicago, Ill.
May	2, 1878	Williamson, D. D., 683 Madison ave., N. Y.
Jan.	6, 1882	Wittmack, Chas. A., School of Mines, N. Y.
March	6, 1879	Ziegler, William, care of Royal Baking Powder Co., 91 Duane st., N. Y.

# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Regular Meeting, Dec. 8, 1882.*

The meeting was called to order at 8.40 P. M. Prof. A. R. Leeds in the chair.

The minutes of the previous meeting were read and approved.

The board of directors had no report. The treasurer stated that we had a balance of about \$200 in the treasury.

The librarian and curator had no report. The committee on endowment fund, stated that the labor of raising the fund was for the present suspended, there being a larger fund in the possession of the society than was needed for the publication of the original papers presented at the present time.

The committee on papers and publications then stated that it had no formal report to present, but trusted that the Society would judge them by the work they had done.

The committee further stated that the November number of journal is in type, and that it will probably be issued the coming week.

The next in order was the election of officers for the ensuing year.

*For President.*—The three following gentlemen received the greatest number of votes: A. R. Leeds, E. R. Squibb, J. C. Booth.

Prof. Leeds declined to be a candidate, on the ground that it was of the greatest importance to the interests of the Society, as a national body, that the honorary office of President should be filled by a non-resident member.

It was then moved that the Society proceed to vote for the two other gentlemen standing highest on the list. This being seconded was duly carried.

Prof. J. C. Booth receiving the greatest number of votes, was duly elected President.

*For Vice Presidents.*—The following gentlemen received the greatest number of votes:

- |                            |                                |
|----------------------------|--------------------------------|
| 1. James H. Stebbins, Jr., | } Three Local Vice-Presidents. |
| 2. A. R. Leeds,            |                                |
| 3. C. F. Chandler,         |                                |
| 4. Arno Behr,              |                                |
| 5. P. Schweitzer.          |                                |
| 6. N. T. Lupton.           |                                |

Mr. Casamajor then moved that the resolution preventing members who have read papers before the Society, from publishing them through any other source except the Journal of the American Chemical Society, for the space of thirty (30) days, be rescinded. This being seconded, was duly carried.

The following gentlemen were then elected to fill the remaining offices of the Society :

*Corresponding Secretary*—P. Casamajor.

*Recording Secretary*—Thomas S. Gladding.

*Treasurer*—T. O'C. Sloane.

*Librarian*—G. A. Prochazka.

*Curators*—William Rupp.

*Committee on Papers and Publications*—E. Waller, C. A. Doremus, L. H. Friedburg.

*Committee on Nominations.*

*Board of Directors.*—It was then moved and seconded that Drs Alsberg and Geyer, be elected Directors, in lieu of C. F. Chandler and James H. Stebbins, Jr.

James H. Stebbins, Jr.,

A. R. Leeds,

C. F. Chandler,

P. Casamajor,

T. S. Gladding,

T. O'C. Sloane,

Geo. A. Prochazka,

E. Waller,

M. Alsberg,

H. Morton,

Wm. E. Geyer,

Wm. M. Habirshaw,

H. Endemann.

Mr. Casamajor then moved that this meeting be declared adjourned till the usual night for conversation. This being seconded was duly carried. After which the meeting adjourned.

James H. Stebbins, Jr.,

Recording Secretary.

At the adjourned meeting, held Dec. 15th, 1882, no quorum being present, official business could not be transacted. A paper on the manufacture of tartaric acid was read by Dr. L. H. Friedburg. Mr. Percy Newman was nominated by E. Waller, Jas. H. Stebbins, Jr., and A. H. Elliott.

# ON THE MANUFACTURE OF TARTARIC ACID.

BY L. H. FRIEDBURG, PH. D.

In this country tartaric acid is hardly manufactured for its own sake but its preparation is unavoidably attached to the manufacture of cream of tartar. Here the starting points for tartaric acid are sablons, waste liquids and residues of different kinds, which render an analytical control troublesome, so that partly because of this, partly because of great dilution, the raw material is treated more or less empirically, after the known and often described methods \* † with chalk and chloride of calcium or gypsum.

Abroad, the manufacture of tartaric acid is not everywhere a mere appendix to cream of tartar manufacturing, but forms an independent branch of manufacture. In such cases the raw material consists either of argols or of dry sablons or of lees. A careful analytical test has to be made before treating either of these mother substances, and the manufacture has to be carried on with the greatest care in order to avoid loss.

Until very recently these tartaric acid factories worked generally after the old plan as indicated above, viz : treatment with chalk and chloride of calcium or gypsum.

But it has to be recorded, and I will briefly do so in the following pages that a very neat and new, patented process, which according to my own experience is commendable, is now also in use.

This process is based on the decomposition of the mother substances, as named above, by slaked lime instead of chalk.

This preparation has been hidden in the European patents ‡ § under the heading, "Methods of obtaining the potassium in the form of hydrate, while making tartaric acid out of argols."

This heading is practically speaking untrue, because, as we shall see later on, the potassium is not finally gained as hydrate, though this is in the course of treatment formed and then transformed into sulphate or chloride.

The chemical process, which takes place in decomposing the bitartrate of potash in any mother substance into tartrate of lime,

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\*Bericht ueber die Entwicklung der chem. Industrie; A. W. Hofmann. Vol. II, page 418, etc.

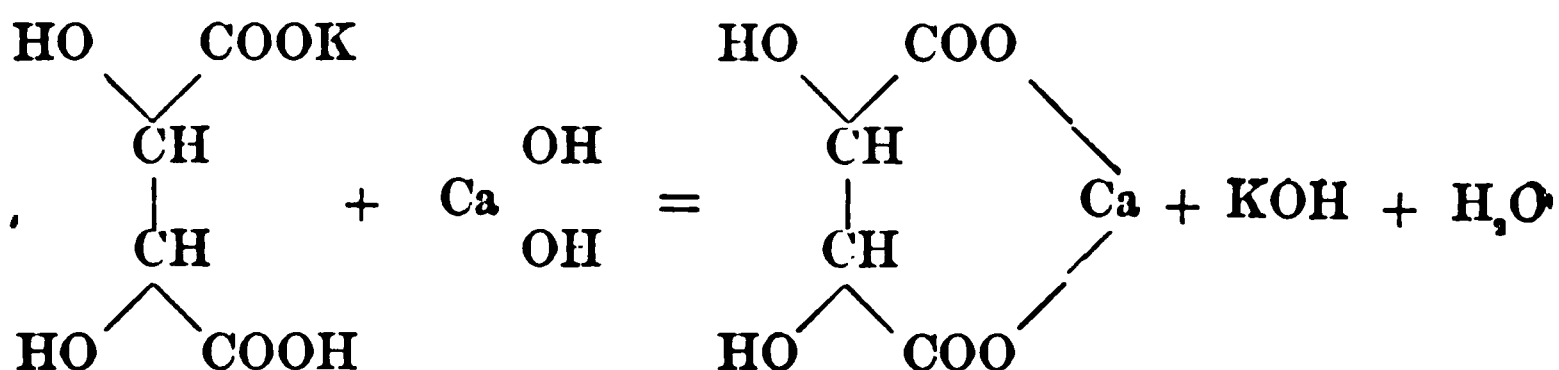
†Journal of the Society of Arts; Robert Warington, Vol. xxiv, No. 1217, page 366, etc.

‡Die chemische Industrie; Dr. Emil Jacobsen; 1879, pages 86 and 238.

§Berichte d. deutschen chemischen Gesellschaft; 1879, page 1366.



by means of slaked lime, is very simply conveyed through the following equation :



The practical difficulty which stood for so long a time in the way of realizing this decomposition for manufacturing purposes, was the difficulty of making the products of decomposition easily filterable. This the patentees have really overcome, and the method of working is smooth and goes like clock work. Slaked lime, freed from coarse pieces is taken in necessary quantities and a milk, not too thin prepared therefrom. This is heated to boiling, and argols, etc., in necessary quantity, are very gradually and in a state of finest powder, introduced into the boiling mass. The charging finished, boiling has to continue for two hours, the condensing steam being enough to keep the mixture in a concentrated form. Hydrate of potash and neutral tartrate of lime are formed in this way. The nitrogenous organic impurities of the raw materials are by the combined action on them, of lime and hydrate of potash, decomposed so as to form ammonia gas which is volatilized with the steam.

Boiling done, which takes place in an iron tank, the mixture is diluted by enough cold water and then the potash is neutralized by either muriatic or sulphuric acid. The process is finished with the help of litmus paper. Here the ammoniacal exhalations are to be considered, so as not to disturb the reaction.

The decomposition as described above takes place under constant stirring by means of an iron stirrer run by machinery.

After the formation of either chloride or sulphate of potash, the mass is still more diluted with cold water and stands best over night, stirring going on continuously, filtration then taking place the next morning. Here filter presses are used to great advantage. It is advisable not to use too high pressure, so as to get a soft cake, which can more easily be washed out, in order to get rid of the mineral potash salts. These latter are either boiled down, as long as the strength of the solution makes it pay, or they might\* be treated with chloride of lime [bleaching powder] and thus trans-

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\*In case chloride of potassium was formed.

formed into chlorate of potash, which I advise manufacturers to try.

The cakes of brown tartrate of lime, which begin to exhale putrid odors by standing too long in a warm place, (in summer time six hours standing often will show this result) have speedily to be decomposed by sulphuric acid. This decomposition takes place in a wooden, lead lined tank, with heavy wooden stirrer moved by steam. The decomposition takes place in the cold and its completion is determined easily by methyl violet test paper.\* No analysis has to be made here, if good paper is at hand, which allows one to guide the reaction so as to get the necessary or the excess of sulphuric acid wished for.

The brown solution of tartaric acid is filtered through filter-presses into wooden receivers.

It is not advisable to evaporate this acid down to the point of crystallization, because it contains impurities enough to spoil the mother liquors at a too early stage. If the course of manufacturing demands a readier transformation of raw material into money, this crude acid solution might be concentrated in the leaden pans to the right concentration for crystallizing or for precipitation by the stirring process, which we shall deal with on another page.

It is preferable to reprecipitate this acid as tartrate of lime, finishing the reaction with chalk and using litmus test paper.

The tartrate of lime thus obtained is filtered on a vacuum filter or by centrifugal power. Of course washing takes place, though slightly. This tartrate of lime is crystalline, light greenish-yellow, keeps perfectly well for any length of time required without decomposing.

It is decomposed in an apparatus similar to the one used for decomposition of the first brown tartrate of lime, by sulphuric acid, in the cold and the reaction finished with the aid of methyl violet test paper. The filtration of the very white gypsum thus obtained cannot be done through filter presses but has to take place on a vacuum filter, very thorough washing being required.

The tartaric acid solution thus obtained ought to stand between 12° and 14° Bé. It is ejected into the lead pans, evaporated at 80° C to the necessary density, by which dissolved gypsum is precipitated, run into the crystallizing boxes and let stand for crystallization. The crop of brown crystals is redissolved to a liquid of the

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\*Journal of the American Chemical Society; T. O'Connor Sloane, Vol. IV, Nos. 1-4, page 31, etc.

density 25° Bé and treated with bone black, which has been purified by muriatic acid, (so as not to leave a trace of phosphates,) at a medium temperature and under stirring.

The discolored liquid is run through a filter-press and thence into special lead pans. It is evaporated down to about 39° to 40° Be and run into lead boxes for crystallization.

The crystallization being a comparatively slow process, this liquid may be run into a proper tank with stirrer, stirred for several hours, thus yielding a crop of small crystals right away.

Either crystals are washed and dried in centrifugals, by using steam for washing.

The liquid running off from the first crystallization yields after evaporation another crop of white crystals. Then it becomes a brown mother liquor.

The mother liquors of the brown crystals can, under careful attention, be carried along through the sixth or seventh crystallization. Then the predomination of sulphuric acid and impurities does not allow further crystallization.

The mother liquors at that stage are diluted to a proper density, the greater part of the sulphuric acid removed by addition of slacked lime milk and the filtered liquid has then principally to be freed from iron salts and from phosphate of alumina.

The iron is easily expelled by taking care to keep it in the form corresponding to the protoxide, the presence of the phosphate of alumina makes it necessary to treat the liquids boiling with milk of lime, thus precipitating phosphate of alumina and forming an acid tartrate of lime, which is soluble. It has to be filtered hot and is decomposed by an addition of sulphuric acid, thus yielding very pure solutions of tartaric acid.

If a transformation of the acid thus gained, into bitartrate of potash should be wished for, which hardly would be prudent, the simplest way of arriving at this end would be the following :

The solution is divided into two equal parts, one-half *saturated* by caustic or carbonate of potash, so as to form a neutral tartrate of potash and then the other half added for the precipitation of the bitartrate.

New York, 15th December, 1882.

ON THE ACTION OF PHTHALIC ANHYDRIDE UPON  
GALLIC ACID.

BY GEORGE A. PROCHAZKA.

Mr. Stebbins\* appears to have entirely misunderstood the gist of my remarks at the November meeting.

More than a year ago I experimented upon the action of phthalic anhydride upon gallic acid, with results similar to those of Mr. Stebbins. My object was to find a more economical method for the production on a large scale of gallein (and coerulëin) than given in the books. The substitution of gallic acid in place of pyrogallol readily suggested itself. Subsequently I found in a synopsis by Ch. Lauth of the report on dye-stuffs, of the jury of the Paris International Exhibition of 1878, (Monit. Teint. 1878) that gallein was prepared by heating together phthalic anhydride and pyrogallic or *gallic* acid. My own results had been anticipated. My own results were never published. In making my remarks at the November meeting it was not my object to substitute a doubtful claim of originality, substantiated by experiments hid under the bushel, in place of Mr. Stebbins,' but to call attention to the fact that he had been anticipated as early at least as 1878 by those European manufacturers, who had utilized the reaction in question. Mr. Stebbins does not appear to have been aware of this fact, which through a small notice had become known only to the initiated few. The very careful and detailed experiments (much more exhaustive than my own) of Mr. Stebbins, merit the widest attention, and the public owes to him a debt of gratitude for their publication.

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\* Journal Am. Chem. Soc. IV., 248.

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# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Regular Meeting January 5, 1883.*

Meeting called to order at 8:50 P. M. Mr. Jas. H. Stebbins, Jr., in the chair. No quorum being present, business was dispensed with.

The following papers were then read by Dr. A. R. Leeds.

1. "On Xylidine-Acrolein."
2. "Cryptidine."
3. "On Oenanthol-Aniline ; Oenanthol-Xylidine, and Oenanthol-Naphthylamine."
4. "On the Products of Distillation of Castor Oil in Partial Vacuo."

After several questions by Dr. Friedberg and Mr. James H. Stebbins, Jr., a paper by F. B. Venable, Ph. D., "On Heptylmalonic and Heptylacetic Acids," was then read.

After proposal of several new members, the meeting adjourned.

THOMAS S. GLADDING, Cor. Secretary.

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## XYLIDINE-ACROLEIN.

BY ALBERT R. LEEDS.

In the Ber: d. D. Chem. Gesell. Vol. XV, p. 1158, I have shown that diphenylamin unites with acrolein to form the compound  $(C_{12}H_{10}N)_2C_3H_4$ , one molecule of water being eliminated. Since that time I have examined the action of acrolein upon xylidine, the method of procedure being somewhat different. The acrolein was distilled directly into an alcoholic solution of xylidine, when a dark red precipitate was formed together with a large amount of a sticky resinous material. This precipitate is soluble in alcohol, ether and bisulphide of carbon. Finding that the resinous by-product was more soluble in dilute alcohol than the principal substance, the attempt was made to purify the latter by repeated washings with alcohol. It failed entirely. The entire removal of the adherent resinous material being impossible by this method.

When to an alcoholic solution of the impure mass, bromine is added in slight excess, a precipitation takes place of what is apparently a bromine compound of xylidine-acrolein. This compound is

soluble in benzole, chloroform, ether and alcohol, insoluble in bisulphide of carbon. It could not be crystallized from any one of these solvents, nor sublimed without decomposition.

Finally, after many unsuccessful trials, the following method was found to afford the xylidine-acrolein in a state of purity. Instead of distilling the acrolein directly into the xylidine, the acrolein in slight excess was added to an alcoholic solution of the latter body. The mixture was then digested for several hours upon a water-bath, until the smell of acrolein had almost entirely disappeared. The resulting mass, which was of a dark red color and extremely sticky, was freed as far as possible from the surrounding liquid, and boiled a number of hours with water. The longer it was boiled with water the less sticky it became, until finally it could be broken into small lumps and removed from the flask. These lumps were then pulverized in a mortar, returned to the flask and boiled with alcohol under a return cooler. This second boiling with alcohol had the result of taking out a large amount of the coloring-matter, but at the same time of making the mass sticky as before. The boiling with water and afterwards with alcohol had therefore to be repeated alternately many times, until at last a product was obtained quite insoluble in alcohol, and which was left hard and brittle after boiling with that solvent.

The purified body, which was of a reddish-yellow color, could not be made to crystallize from any solvent, nor did it yield crystallizable derivatives. As before stated, bromine enters into combination with it with great energy. Nitric acid converts it into a pastry mass that cannot be sublimed or crystallized.

The analysis gave N 8.49 per cent., (theoretical 8.8 per cent.) and showed that the substance was xylidine-acrolein, found according to the equation.



## CENANTHOL-ANILINE, CENANTHOL-XYLIDINE AND CENANTHOL-NAPHTHYLAMINE.

BY ALBERT R. LEEDS.

Cenanthol was prepared from castor oil by heating the oil in partial vacuo at a temperature of about 150°. The flask containing the castor oil was connected with a Liebig's condenser. As the volatile substances were distilled off they were condensed and



collected in a receiver connected with a Bunsen pump. The ænanthol was purified by subjecting the distillate to a second distillation and collecting that portion that came over at  $154^{\circ}$ . After two or three such treatments the ænanthol was obtained in a state of purity sufficient for the purposes contemplated in this article.

In preparing the following compounds molecular proportions were in each case used. 70 grms. of ænanthol and 57 grms. of aniline were gradually mixed together. The mixture became very hot; the temperature rising from  $27^{\circ}$  to  $89^{\circ}$ . The resulting liquid mass is very mobile, much more so than either aniline or ænanthol.

In making the ænanthol-xylydine 70 grms. of ænanthol were added to 74 grms. of xylydine. The temperature rose from  $27^{\circ}$  to  $85^{\circ}$ , and the resulting product was also extremely mobile.

70 grms. of ænanthol were poured on to 88 grms. of naphthylamine.

The latter melted very rapidly and after combination had taken place the temperature of the liquid had risen to  $75^{\circ}$ .

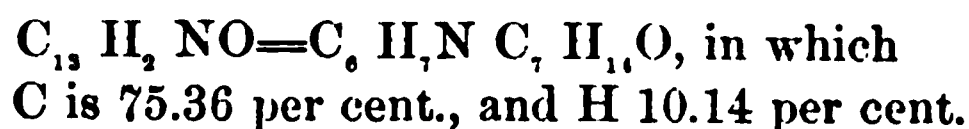
All the three compounds were heated on the water bath under the return cooler for six hours to ensure complete combination. At the end of that time the flasks containing the three portions were removed and small portions taken from each which were subjected to distillation. The distillate in each case consisted of an uncombined base and some product of decomposition. The method of purification by distillation was thus far abandoned.

It was next found that the substances could not be purified by any solvent, since the ænanthol-aniline, ænanthol-xylydine, and ænanthol-naphthylamine are as soluble in alcohol, ether, benzol, chloroform and carbon bisulphide, as their respective bases.

Eventually the most suitable process proved to be the following: Each of the three compounds was dissolved in about 150 grms. of glacial acetic acid and heated several hours on the water bath to ensure complete combination between the acetic acid and the excess of the bases aniline, xylydine, and naphthylamine. When the bases were completely changed into their respective acetates, a large excess of water was added which threw down the ænanthol-aniline, etc., while the acetates remained in solution. The precipitated substances were thoroughly washed with water until every trace of acetic acid had disappeared. The three compounds were then dried at a temperature of  $100^{\circ}$ .

A portion of each of the three compounds was preserved for analysis while the rest was subjected to distillation.

The œnanthol-aniline as finally purified is a reddish-colored mobile liquid, having an agreeable ethereal odor, which resembles neither aniline or œnanthol. Its analysis yielded C 75.10 per cent., H 10.28 per cent., corresponding to the formula,



The œnanthol-xylylidine closely resembles œnanthol-aniline in appearance and smell. Its formula was found to be



	Found.	Calculated.
C.....	76.94	76.59
H.....	10.00	10.64
N.....	5.93	5.96

The œnanthol-naphthylamine resembles the two preceding compounds in its appearance and properties, but was still more pronounced in its ethereal odor, which resembled that of pineapple.

Its formula is  $\text{C}_{17} \text{H}_{23} \text{NO}=\text{C}_{10} \text{H}_9 \text{N C}_7 \text{H}_{14} \text{O}.$

	Found.	Calculated.
C ..	79.03 per cent.	79.38 per cent.
H.....	9.50 “	8.95 “

It will be noted that all these compounds formed synthetically by the direct union of one molecule of œnanthol with one molecule of the aromatic base, no water being eliminated, have a correspondingly elevated heat of combination. They are stable bodies, and were capable of sublimation, with only partial decomposition. The sublimes were not crystalline, and were identical in physical and chemical properties with the original substances, yielding on analysis the same formulæ.

## SOLID RESIDUE FROM THE DISTILLATION OF CASTOR OIL IN VACUO.

BY ALBERT R. LEEDS.

In making œnanthol from castor oil there is always left in the flask a highly elastic, sticky substance, which was first investigated by Staněk (*Jour. Pr. Chem.*, Vol. 63, p. 138.) Although Staněk's results have never been accepted as conclusive, yet no one has reinvestigated the nature of this body, for which reasons I was induced to make the following research.

According to the recommendation of Krafft, Ber. Ber., Vol. x, p. 2034, the œnanthol was prepared by distilling castor oil in vacuo a pressure of about 100 m. m. being maintained. The operation must be watched very closely towards the end, because the residue in the retort, as soon as all the œnanthol has been driven off, liberates a large amount of gas which causes the caoutchouc-like mass to swell enormously and to fill the whole flask. Therefore, as soon as the bubbles of gas begin to appear, the vacuum is instantly relieved and the lamp removed. The residue, on the liberation of the gas, changes from a comparatively thin liquid to a very viscid mass that finally, on cooling, has almost the consistency of caoutchouc. It could not be entirely removed from the flask by mechanical means, and therefore, after as much as possible had been removed by a spatula, the remainder was saponified either with aqueous or alcoholic potash.

The caoutchouc-like mass was thoroughly washed, first with alcohol and then with ether, in neither of which is it at all soluble. Before washing, the substance is sticky and elastic, but the ether and alcohol remove all traces of stickiness, and most of the elasticity disappears. Long standing has the same effect, but in a lesser degree. The original color of the substance is a brownish-red, but after washing it becomes grayish-white with a very faint tinge of yellow. The analyses were made on the body thus purified.

In his analysis Staněk gives the formula  $C_{42}H_{66}O_6$  as calculated from C 77.11 per cent. H 10.77 per cent. and O 12.12 per cent. The percentages as found by me were : C 76.47 per cent. H 10.69 per cent. and O 12.84 per cent.

The caoutchouc-like body by long continued boiling with aqueous or alcoholic potash is completely saponified. All the residues obtained were saponified, the solutions filtered hot, and the filtrates decomposed by hydrochloric acid. The organic acid in combination with potash, after decomposition by a mineral acid, presents none of the characteristics of the original caoutchouc-like mass. On the contrary, it is an oily body lighter than water, and has a reddish-brown color. The oil is extremely soluble in alcohol and ether, and insoluble in water. It was purified for analysis by dissolving in ether, after repeatedly washing with hot water, filtering, evaporating off the ether and drying at 100°.

The purified oil on analysis gave : C 70.22 per cent, H 11.04 per cent. O 18.74 per cent. Staněk in his analysis of the same oil, prepared by a slightly different method, found C 70.40 per cent, H

11.00 per cent, O 18.60 per cent, from which he deduces the formula  $C_{36}H_{66}O_7$ .

In the acid filtrate from the oil decomposed by hydrochloric acid, no glycerine could be found, but only a small amount of a resinous body which was not investigated. This result also corresponds with that obtained by Staněk.

A considerable amount of the oil was subjected to fractional distillation. When heated, the oil turns black and is decomposed, yielding at ever-increasing temperatures a distillate of light and almost colorless oils that have a very pungent and peculiar though not a disagreeable odor. These oils come off at points ranging from  $110^\circ$  to  $250^\circ$ . They were collected in three separate portions, having boiling points from  $110^\circ$   $150^\circ$   $160^\circ$   $200^\circ$  and above  $200^\circ$ . Each portion was distilled three times, and finally three were taken whose boiling points were  $120^\circ$   $180^\circ$  and  $220^\circ$  C.

Their analysis yielded :

	120°		180°		220°
Carbon.....	68.20	.....	68.89	.....	68.85
Hydrogen.....	11.29	.....	12.60	.....	11.50
Oxygen.....	20.51	.....	18.51	.....	19.65

These oils do not form salts with potash or soda. On exposure to light and air they turn darker from absorption of oxygen.

Nitrogen was sought for in all of the oily products of the caoutchouc-like substance, but its presence in any could not be established.

The results above given do not bear out the views of Staněk with regard to the constitution of the caoutchouc body and its derivatives. Staněk assigns the formula  $C_{42}H_{66}O_6$  to the caoutchouc body, and terms it an Acryloxide compound consisting of his Pyroricinic Acid and Acrolein.



In this case the compound ought to contain Acrolein which it does not. No Acrolein could be detected either in the process of saponification or of distillation.

In the next place, the Pyroricinic Acid of Staněk which he regards as a fatty acid, although it has no analogies to any known series of fatty acids, is first formulated as  $C_{36}H_{66}O_7$ . It was made by converting the oil obtained by saponification of the caoutchouc body, into a lead salt. But this body after keeping for three days at a temperature of  $100^\circ$ , became (according to Staněk).  $C_{36}H_{66}O_7$ . He furthermore states that the water which thus came off did not

exist in the original acid in the form of water, inasmuch as the correct formula of this acid should be regarded as  $C_{16}H_{22}O_4$ .

I have not attempted to give rational formulæ for the caoutcho u body or for its oily distillates. I have merely given these results the obtaining of which involved very considerable labor on my, own part and that of my assistant Dr. Edgar Everhart, in the hope that some one will repeat the work with such large quantities of material as eventually to obtain by fractional distillation or otherwise, homogenous products.

---

## CRYPTIDINE.

BY ALBERT R. LEEDS.

When xylidine-acrolein, prepared as stated in a preceding article was subjected to dry distillation some oily drops of peculiar smell were given off. A considerable amount of this substance, therefore, after careful purification was thoroughly dried at a temperature of  $110^{\circ}$  and finely pulverized. Small portions of about 20 grms. each were introduced into a small tubulated retort, placed in an air-bath and subjected to distillation, carried on very slowly to avoid carbonization. No record could be taken of the temperature of the distillation since it exceeded  $360^{\circ}$ . At first, before decomposition set in, a small amount of water came over which was of a slightly acid reaction, and which was rejected. When the temperature rose higher and decomposition began, a distillate composed of oily drops mixed with water of an alkaline reaction, came over. The residue in the retort consisted of a porous carbonaceous mass, extremely difficult of combustion. The alkalinity of the distillate, was due to free ammonia given off in considerable quantities during the operation, the retort at the end being filled with its fumes.

About 155 grms. of the xylidine-acrolein were thus distilled in small portions at a time and the distillates collected. The water was separated from the oil by filtering through a wet filter, and heated for several hours at a temperature of  $100^{\circ}$  till the weight was constant. The yield from 155 grms. of the xylidine-acrolein was 11 grms., or a little over 7 per cent.

Several unsuccessful attempts were made to purify the distillate, but no constant boiling point could be obtained. A small amount

of carbonaceous residue was left in the distilling flask at the close of each experiment.

The crude oil has a disagreeable smell and a very bitter taste. It forms crystalline salts with acids; sulphuric, hydrochloric, acetic, etc. Finally, the best method of purification was found to be that of decomposing its hydrochloric acid salt by means of alkali. The mother liquor, obtained by treating the oil with hydrochloric acid and consisting of a bad-smelling, thick liquid, was drained off from the crystals, and these were further cleaned by pressing between filter paper. The crystals were then dissolved in a small amount of water and filtered from an insoluble oily scum. The aqueous solution was allowed to crystallize, and the crystals were treated in the same way until quite pure. Finally the aqueous solution of the salt was decomposed with a little caustic potash. The precipitated oil was freed from the alkali, by repeated washing with water, filtered and thoroughly dried at 100°. The dried and purified oil was redistilled at 270° a boiling point that remained constant. It is reddish yellow in color, and possesses a disagreeable odor.

The final analysis was made on this product and gave a formula corresponding to cryptidine,  $C_{11}H_{11}N$

	Found	Calculated
C .....	84.45 pct.	84.08 pct.
H .....	7.38 pct.	7.00 pct.
N .....	8.99 pct.	8.92 pct.

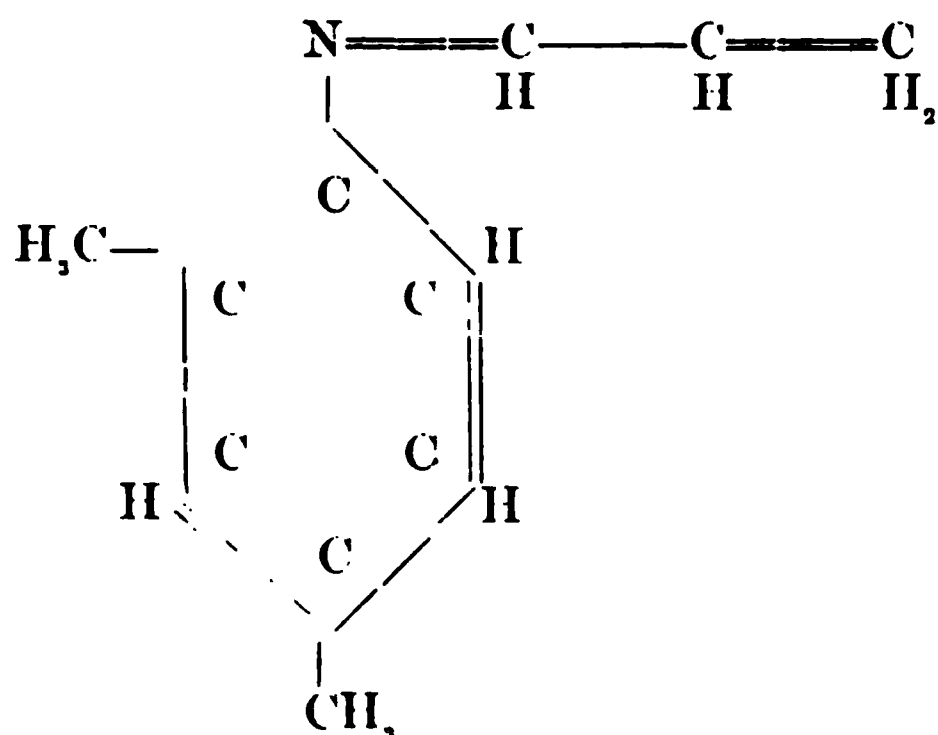
The hydrochlorate crystallizes in fine thin, colorless, tubular crystals which may, by careful heating, be sublimed, although a partial decomposition takes place. On analysis it yielded 18.04 per cent. Cl, theory 18.34 per cent. required.

When to an aqueous solution of the hydrochlorate a solution of platonic chloride is added, there is instantly a precipitation of a fine yellow crystalline compound of the double salt of cryptidine and platonic chloride. The double salt is soluble in water, but is precipitated on the addition of platonic chloride in excess. It was purified by washing with alcohol in which it is insoluble and crystallizing from water.

It will be noted that this is the first successful attempt to form cryptidine synthetically, and that it likewise yields the oil directly and in a state of purity.

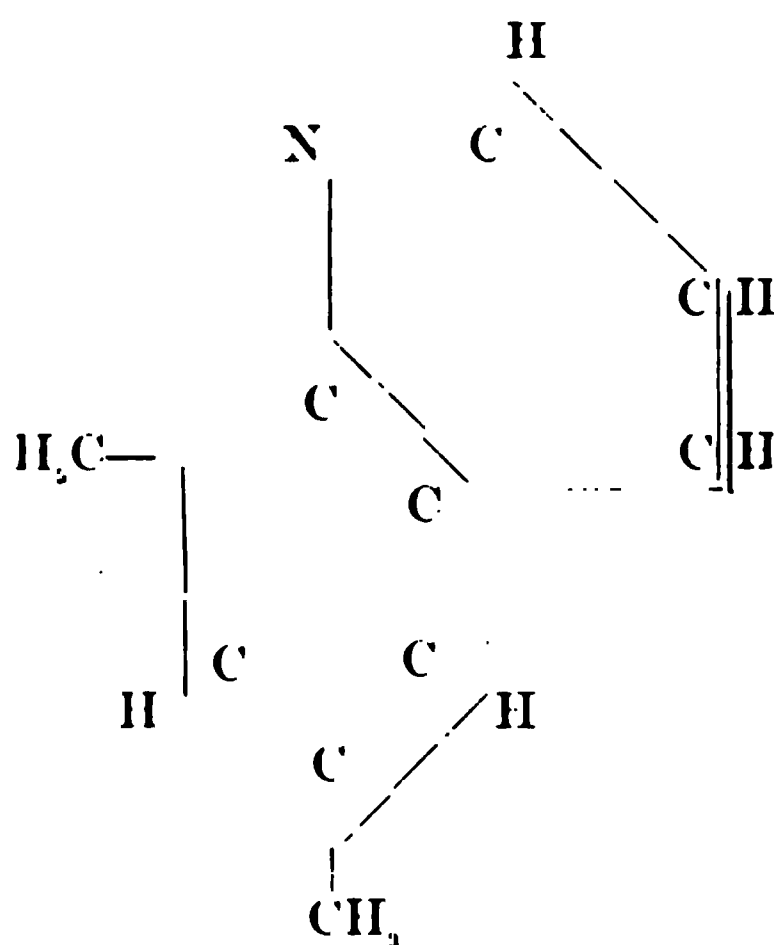
This synthetic formation of cryptidine, affords a valuable opportunity of obtaining an insight into its probable structural con-

stitution. The constitution of xylidene-acrolein, it was the meta compound which was employed, might be represented as



The oxygen of the acrolein is represented as combining with the two atoms of the nitrogen, in order to account for the fact that the metaxylidene-acrolein is entirely devoid of basic properties, and cannot be made to form salts. On heating, two atoms of hydrogen are given off, one from the extremity of the acrolein chain the other from the benzene ring. This being the case, the extremity of the chain is brought round to form by the union of the bonds thus opened another ring.

In other words the Cryptidine would contain the benzene ring, and a side ring with the N atom occupying the usual position of the C atom, the bonds being probably distributed as in the annexed diagram.



## HEPTYLMALONIC AND HEPTYLACETIC ACIDS.

F. P. VENABLE, Ph. D.

A brief mention of the preparation of these two acids has already been made (Ber. XIII., 1657). I have since utilized the small amount of the acids which I had on hand in the preparation of some new salts, and will give a description of them, as they serve to characterize these acids more fully than was done in the previous notice.

HEPTYLMALONIC ACID  $C_{10}H_{18}O_4$ .

This acid belongs to the oxalic acid series and is an isomer of sebacic acid. It can be prepared from heptylmalonic ether. The simplest decomposition of this ether by means of an alkali is of course the breaking up into alcohol and heptylmalonic acid. We can look upon the reaction as taking place in accordance with the following formula :



An alcoholic solution of potassium hydroxide was used. A small portion of the ether and four times the theoretical amount of potassium hydroxide were heated four to six hours in a small flask upon the water bath. At the close of the reaction water was added, and then hydrochloric acid, and the whole gently heated. The heptylmalonic acid separated as a brown-colored oil on the surface of the water. This was then shaken with ether, allowed to stand for evaporation of the ether and the residue placed in a desiccator over sulphuric acid. The mass solidified, but was still a yellow color, and after drying gave the melting point at 90–93° C. In order to free the acid from all impurities it was washed well with petroleum ether. It proved almost insoluble in this liquid, and was left as a white crystalline mass, melting by 97–98° C. (uncorrected). It is but slightly soluble in water, but easily so in alcohol, chloroform and ether. The solution has a strongly acid reaction. Analyses were made as follows :

I. 0.1432 grm. of acid taken	% C=59.76	% H=9.01.
II. 0.1557 grm. of acid taken	% C=59.59	% H=9.04.
Calculated for $C_{10}H_{18}O_4$	% C=59.41	% H=8.91, % O=31.98.

The acid is dibasic and capable of substituting carbonic acid in the cold. Heated to 130–160° C. it breaks up into heptylacetic



acid and carbon dioxide. The salts crystallize very indistinctly, if at all, and are easily soluble only in the case of the alkalis.

#### SILVER HEPTYLMALONATE.

An aqueous solution of ammonia was added to the acid until nearly neutralized, and the ammonium heptylmalonate formed in this way was added to a solution of silver nitrate, causing a white precipitate of silver heptylmalonate. The precipitate is heavy and white, seemingly composed of minute, indistinct crystals, and is quite insoluble in water, whether cold or hot. Alcohol also fails to dissolve it appreciably. It was dried in a steam-bath to a constant weight. Only after long keeping does it change to a reddish brown. The melting point, as determined with a portion only slightly changed in color, was  $244^{\circ}\text{C}$ . Two determinations of silver were made.

- I. 0.4330 gram. silver salt taken, % Ag=51.34.
- II. 0.3904 gram. silver salt taken, % Ag=51.56.
- Calculated for  $\text{C}_{10}\text{H}_{16}\text{O}_4\text{Ag}_2$ . % Ag=51.92.

#### BARIUM HEPTYLMALONATE.

As in the case of the silver salt, this was obtained as a precipitate from barium chloride by means of ammonium heptylmalonate. It is a white amorphous powder, insoluble in water and alcohol. The salt was dried at  $100^{\circ}\text{C}$ . and analyzed. It can be heated to  $160^{\circ}\text{C}$ . without suffering decomposition.

- I. 0.3002 gram. barium salt taken, found % Ba=40.19.
- II. 0.5133 gram. taken, found % Ba=40.45.
- Calculated for  $\text{C}_{10}\text{H}_{16}\text{O}_4\text{Ba}$ . % Ba=40.65.

#### COPPER HEPTYLMALONATE.

The ammonium salt was not used in the preparation of this compound, but the acid itself (slightly impure) dissolved in water and added to a concentrated solution of pure cupric sulphate. The impure acid was used because the traces of impurity made it much more soluble in water, and the nature of the impurity did not seem to interfere with the formation of the cupric salt. The addition of the acid caused a light-blue precipitate, which was washed well with water. As it was somewhat soluble in water, an excessive use of this had to be avoided. It is more soluble in alcohol, and on evaporation is left behind as an indistinctly crystalline mass. On standing several days over sulphuric acid it becomes almost dry

and loses at 100° C. the last traces of water. On further heating it suffers no loss in weight, until at quite a high temperature it begins to turn brown and decompose. It seems therefore to contain no water of crystallization.

Analysis :

0.3253 grm. taken, found % Cu=23.80.

Calculated for  $C_{10}H_{16}O_4Cu$ . % Cu=24.04.

#### LEAD HEPTYLMALONATE.

Alcoholic solutions of heptylmalonic acid and lead acetate were made, and on mixing them a heavy white precipitate formed. This precipitate was repeatedly washed with water, and some of the last washings were evaporated, yielding scarcely any residue, thus showing the almost entire insolubility of the lead heptylmalonate in that solvent. In alcohol it is slightly soluble. After thorough washing, the precipitate was dried in the steam-bath; drying at 110° C. caused no further loss in weight. A portion of the dried salt was taken for a determination of the melting point; melting at 235° C. The remainder of the precipitate, amounting to nearly 0.2 grms, was taken for analysis. It was dissolved in dilute nitric acid and the lead determined by evaporation with sulphuric acid.

Analysis :

found, % Pb=50.49.

Calculated for  $C_{10}H_{16}O_4Pb$ , % Pb=50.89.

#### ZINC HEPTYLMALONATE.

On adding an alcoholic solution of the acid to one of zinc acetate the whole mass gelatinized, very much like silicic acid. It is stiff and can be cut or broken up with the stirring rod. This jelly-like mass was first washed with alcohol and then boiled with water, becoming compacted, white and flaky and easy to wash. It is somewhat soluble in water when in this form, more so in hot than in cold, separating from a hot solution on cooling in minute crystals. Some of these crystals dried on filter paper were found to melt at 247° C. The rest of the precipitate was collected in a crucible, dried on water-bath and weighed. On heating at a higher temperature, 140°–170° C., slight loss in weight was noticed. This loss was continuous, and resulted from commencing decomposition. The exact temperature at which the decomposition commenced was not determined. The salt is also easily soluble in ammonium hydrate.

The zinc was determined by dissolving in dilute hydrochloric acid and precipitation as carbonate.

Analysis :

- I. 0.1298 grm. salt taken, found % Zn=24.49.
- II. 0.0411 grm. salt taken, found % Zn=24.33.
- Calculated for  $C_{10}H_{18}O_4Zn$  % Zn=24.52.

HEPTYLACETIC ACID,  $C_9H_{18}O_2$ .

According to the researches of Wisliceuns (Annalen, 190.267), ethers of the aceto-acetic series can undergo two different decompositions on treatment with alkalis—one resulting in the formation of alcohol, potassium carbonate and a ketone, the second in the formation of alcohol, potassium acetate and a mono-basic acid. In preparing heptylacetic acid from the heptylaceto acetic ether the methods of Wisliceuns were closely followed, yet it was found impossible to bring about any one decomposition unaccompanied by a partial decomposition in accordance with the second formula. In preparing the acid then, traces of methylactyl ketone were formed at the same time. The mode of preparation was as follows. Four times the theoretical amount of a concentrated alcoholic solution of potassium hydroxide was added to the heptylaceto-acetic ether placed in a flask connected with an inclined condenser, and the mixture heated six hours upon the water bath. The main portion of the alcohol was then distilled off, dilute hydrochloric acid added to the solution left behind, and the heptylacetic acid separated immediately as a brownish thick oil on the surface of the liquid. This oil was washed with water and dried over calcium chloride. As the heptylacetic ether could be procured with difficulty, and the product of acid from its decomposition was very unsatisfactory, it was found more convenient to prepare the acid from the heptylmalonic acid already mentioned.

As is well known, all diatomic, dibasic fatty acids, which have the two carboxyles joined to the same carbon atom, fall on heating into carbon dioxide and a monatomic, monobasic acid. Thus oxalic acid (where the two carboxyls are united to one another) yields on rapid heating carbon dioxide and formic acid. Malonic acid yields carbon dioxide and acetic acid. Heptylmalonic acid, then, should yield carbon dioxide and heptylacetic acid. The heating was done in a small flask with condenser inclined and its open end connected with a pipette which just dipped beneath the surface of some water

in a beaker. In this way the evolution of carbon dioxide, and hence the progress of the reaction, could be watched. The flask was heated in a bath of paraffine. The heptylmalonic acid first melted, then at about  $130^{\circ}\text{C}$  the evolution of carbon dioxide commenced, at  $150^{\circ}\text{C}$  the evolution was quite rapid. The temperature was finally raised to  $170^{\circ}\text{C}$  to complete the reaction, the condenser was then inverted and the dark oily fluid left in the flask fractionated. Up to  $220^{\circ}\text{C}$  nothing, distilled over, then a few drops to  $228^{\circ}\text{C}$ , and the greater part of the acid came over at  $232\text{--}233^{\circ}\text{C}$ . The pure acid was found to boil at  $233^{\circ}\text{C}$ . It is a colorless, oily liquid, with somewhat of the characteristic smell of the acetic ethers, though not so pleasant. It is nearly insoluble in water, but easily soluble in alcohol and ether. Cooled to  $23^{\circ}\text{C}$ , it becomes viscous, but does not solidify. Analyses were made as follows:

Analyses:

I. 0.2033 grm. acid taken, found	% C = 67.97 ;	% H = 11.64.
II. 0.1116 grm. acid taken, found	% C = 68.11 ;	% H = 11.70.
Calculated for $\text{C}_8\text{H}_{16}\text{O}_2$ acid taken	% C = 68.35 ;	% H = 11.40.

This acid is an isomer of pelargonic acid, and is monobasic. Towards litmus its reaction is faintly acid, and only by aid of heat is it capable of decomposing carbonates. With the metals it forms salts which are, as a rule, non-crystalline and difficultly soluble in water. They are fairly stable when heated, decomposition not taking place generally until a temperature considerably above  $100^{\circ}\text{C}$ . has been reached.

#### CALCIUM HEPTYLACETATE.

It was found best to make this salt first and use it in preparing others which were deemed of value in characterizing the acid. Heptylacetic acid was suspended in water and boiled with an excess of calcium carbonate. Bubbles of carbon dioxide were noticed, but the action was slow. The filtered solution, smelling strongly still of the acid, was placed over sulphuric acid in a desiccator. After some days a crust formed over the surface, but no crystals were observed. By further evaporation a white powder was obtained if a good deal of the salt was present, otherwise a dry transparent crust. This was thoroughly dried at  $100^{\circ}\text{C}$ . The salt is not very soluble in water, nor in alcohol. Hydrochloric acid dissolves it with the separation of the acid. The analysis was carried out by simple ignition.

## Analysis :

0.1181 grm. salt taken ; found	% Ca = 11.38.
Calculated for $(C_7H_{17}O_2)_2Ca$	% Ca = 11.29.

## SILVER HEPTYLACETATE

was gotten as a heavy white precipitate by adding silver nitrate to the aqueous solution of the calcium salt. This was filtered off and washed well with water, alcohol, and finally ether, and dried in the steam-bath. It is nearly insoluble in cold water, very slightly more soluble in hot, and not perceptibly soluble in either alcohol or ether. There was no loss in weight nor change of color from heating in the steam-bath, nor in the air-bath as high as 130° C. Between 200° and 220° C complete decomposition takes place. Long continued exposure to the light changes the color, though not very deeply.

## Analysis :

I. 0.3065 grm. salt taken ; found	% Ag = 40.88.
II. 0.2910 grm. salt taken ; found	% Ag = 40.65.
Calculated for $C_7H_{17}O_2 Ag$	% Ag = 40.76.

## BARIUM HEPTYLACETATE.

This salt was gotten by the direct action of the acid, suspended in water, on barium carbonate. It is somewhat soluble in water and rather more soluble in alcohol. A saturated aqueous solution remained some days over sulphuric acid in a desiccator from which most of the air had been withdrawn, but no crystals were obtained.

The solution was then evaporated to dryness, and the white powder left heated in an air bath to 150° C. without loss of weight. In determining the barium the salt was slowly and carefully tested till the residue was nearly white, ammonium carbonate was then added and the whole again heated until a constant weight was obtained.

## Analyses :

I. 0.3042 grm. salt taken ; found	% Ba. = 30.13.
II. 0.1970 grm. salt taken ; found	% Ba. = 29.55.
Calculated for $(C_7H_{17}O_2)_2Ba$ .	% Ba. = 30.38.

## COPPER HEPTYLACETATE.

To prepare this ammonium heptylacetate was first made by treating the acid with an aqueous solution of ammonia. The solution of this was then added to a cupric sulphate solution and the greenish-

blue precipitate, gotten in this way, washed well with water. It was then dissolved in alcohol and after slow evaporation of the alcohol there was left instead of crystals a few deep green oily drops on the bottom of the dish. Minute crystals could be gotten however, in the form of a precipitate by slowly adding water to the alcoholic solution. An analysis showed the purity of the compound.

Analysis :

0.2262 grm. salt taken ; found % Cu = 16.53.

Calculated for  $(C_7H_{17}O_2)_2Cu$ . % Cu = 16.78.

ZINC HEPTYLACETATE.

The first mode of preparation attempted was by adding a solution of calcium heptylacetate to one of zinc acetate. This gave a white cloudy precipitate which settled slowly. It was washed with water and then with alcohol. It seemed somewhat more soluble in alcohol than water, though not very soluble in either. On drying in the steam-bath after washing with alcohol, a white sticky mass was left, continued drying at  $100^{\circ} C$ . however, caused no further loss in weight. The analysis made of this precipitate showed only an approximate purity, the percentage of zinc being too high. A different mode of preparation was then tried. The acid, suspended in water, was boiled with freshly precipitated zinc carbonate. A slight solution of carbon dioxide was noticed, an excess of acid was added and the boiling was long continued. Analysis of the product finally showed by the high percentage of zinc, that much of the zinc carbonate remained unacted upon. A last attempt with the small remaining portion of the acid was made by dissolving it in alcohol and adding to it an alcoholic solution of zinc acetate. This caused a slight turbidity only, as both the acetate and the heptylacetate of zinc are soluble in alcohol. The acetate, however, is more soluble in water than in alcohol, just the reverse being true, apparently, for the heptylacetate. On adding water, then, to this solution of the two in alcohol, a cloudy white, slowly settling precipitate was gotten. The amount however was too minute for any reliance to be placed upon an analysis, hence none was attempted.

The ammonium salt and the potassium salt of this acid was made incidentally for the preparation of other compounds by saturating the acid with solutions of ammonium and potassium hydroxide respectively. They seemed easily soluble in water and in alcohol, but gave no good crystals and were not analyzed.

## ABSTRACTS.

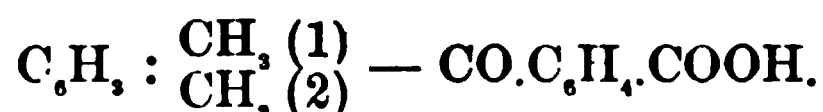
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Abstracts from *Berichte der Deutschen Chemischen Gesellschaft*, by Percy Neymann, Ph. B.

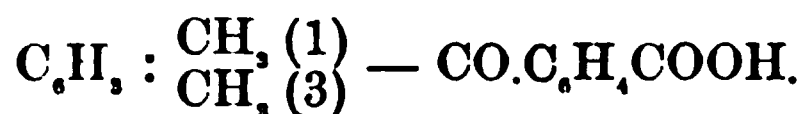
**The Action of Sodium on Lupinin.** G. BAUMERT. (Vol. XV., p. 631).—Partly controversial. From a series of experiments it is shown that when sodium acts on lupinin a sodium substitution product is formed, which, in contact with water, exchanges the sodium for hydrogen, hence acting like a sodium alcoholate.

**Anhydrolupinin.** G. BAUMERT. (Vol. XV., p. 634). Anhydrolupinin was produced by the action of phosphoric acid anhydride and lupinin chloride, by heating five to six hours to about 190° C.

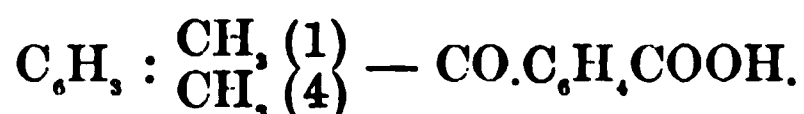
**On the Acids produced from Xylols and Phtalic Acid Anhydride.** FRANZ MEYER. (Vol. XV., p. 636). The new acids may be regarded as hydrocarbons, into which the group  $\text{COOH.C}_6\text{H}_4\text{.CO}$  has been introduced. Orthoxylal-phtaloylic acid from ortho-xylol and phtalic acid anhydride has the formula :



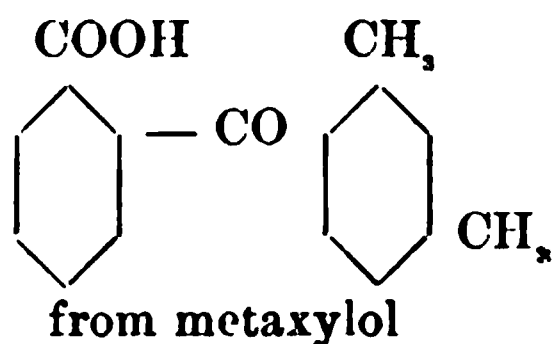
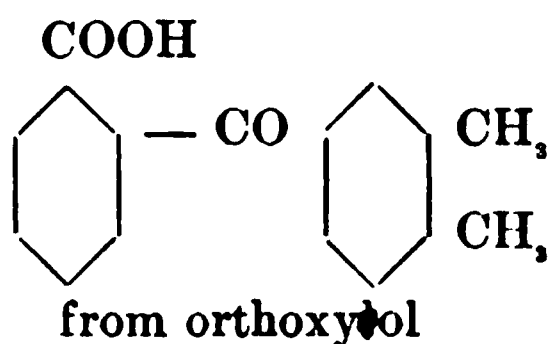
Metaxylol-phtaloylic-acid from one part of phtalic acid anhydride to three of metaxylol in presence of one and one-half parts of aluminium chloride has the formula :

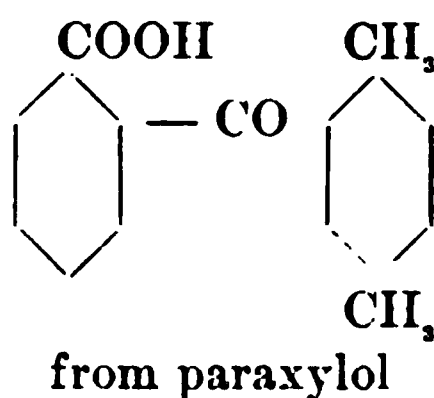


Paraxylol-phtaloylic-acid from paraxylol and phtalic acid anhydride has the formula :

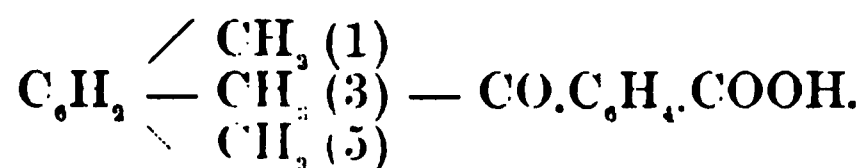


The constitutional formulæ of the three acids would hence be :





**On Mesitylenphtaloylic Acid.** J. GRESLY and F. MEYER. (Vol. XV., p. 639).



The action of phtalic acid anhydride on mesitylen in presence of chloride of aluminium is analagous to that on metaxyl. Analyses correspond to the formula  $\text{C}_{17}\text{H}_{16}\text{O}_3$ .

**On the preparation of Aromatic Phosphoric Acid Ethers.** A. WEBER and R. HEIM. (Vol. XV., p. 639). Phenols can be converted into amines and into the aromatic ethers, but the substitution of the hydroxyl group is often found to be very difficult. The authors find that by means of phosphorous oxychloride the ethers can be formed with comparative ease.

**On the action of Acetylchloride and Glacial Acetic Acid on Fumaric Acid and on the Decomposition of the Mono-substituted Succinic Acid Anhydrides.** R. ANSCHUTZ and C. BENNERT. (Vol. XV., p. 640).—14 grms. of acetylchloride, 28 grms. of glacial acetic acid, and 10 grms. of fumaric acid, when permitted to act upon each other for twelve hours, give a clear, light yellow liquid which, when subjected under ordinary pressure to distillation separates hydrochloric acid copiously, and maleïnic acid anhydride distills over. If the distillation is performed under diminished pressure (11 to 12 mm. mercury) acetyl-chloride, acetic acid and acetic acid anhydride distill over at first; at  $80^\circ$  maleïnic acid anhydride begins to come over until at  $125^\circ$  to  $126^\circ$  a colorless liquid distills over, which becomes hard. This body was found by analysis to be monochlorsuccinic acid anhydride. Acetylchloride and glacial acetic acid react upon each other in such a manner when boiled that by the evolution of hydrochloric acid acetic anhydride is formed. The hydrochloric acid attaches itself to the fumaric acid and forms monochlorsuccinic acid. The latter



is converted into monochlor-succinic acid anhydride by acetyl-chloride.

**On Oxalines and Glyoxalines.** O. WALLACH. (Vol. XV., p. 644).—Preparation and description of methylglyoxaline, propylglyoxaline, amylglyoxaline, and some of their salts.

**On the action of Acid Chlorides and Bromides on Chinones.** H. SCHULZ. (Vol. XV., p. 652).—The experiments gave reason for the conclusion that acid chlorides and bromides act in a similar manner on chinones as hydrochloric and hydrobromic acids; chloridized or bromidized hydrochinonethers are formed. It must be assumed that the respective chlorine or bromine anhydrides split up into the free halogen and the acid radical; the latter attaches itself to the chinon, and hydrogen atoms of the generated hydrochinonether are substituted by the free halogen.

**On the production of active Oxygen.** MORITZ TRAUBE. (Vol. XV., p. 659).

**On Condensation Products of Aromatic Bases.** OTTO FISCHER. (Vol. XV., p. 676).—The aldehydes were made to act either upon the sulphuric or phosphoric acid salts of the aromatic amines in place of, as heretofore, upon the hydrochloric acid salts. The contents of the article give more contributions to the manifold condensations of aldehydes and bases.

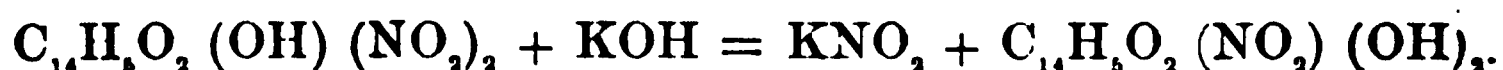
**Note on Alpha-oxychinolin.** KARL BEDALL and OTTO FISCHER. (Vol. XV., p. 683).

**On Basic Acetate of Magnesium.** W. KUBEL. (Vol. XV., p. 684).—Acetate of magnesium when warmed with an excess of magnesia in aqueous solution is converted into the basic salt in the same manner as lead. The basic acetate of magnesium is highly antiseptic, disinfecting, and especially deodorizing. A solution of basic acetate, somewhat cloudy from the presence of basic magnesium acetate, is known in commerce as sinodor.

**On the Compounds of Naphtochinon with Toluidin and Ethylanilin.** LEO ELSBACH. (Vol. XV., p. 685).—The ideas of Liebermann regarding the conversion of beta-naphto-chinon-anilid into alpha-naphto-chinon-anilid gave reason to the supposition that other aromatic aminebases could react in a similar manner on beta and alpha-oxynaphtachinon, and that two series of amid compounds

would be formed by the two chinones. The author experimented with para-toluidin and the two naphtho-chinones and oxynaphtho-chinones, and verified the supposition completely. Orthotoliudin was tried in the same manner, but the operations were performed with much greater difficulty. Experiments with an imid base also proved the correctness of the constitution formula set up by Liebermann.

**On a new method of preparing Alizarin-Orange.** S. E. SIMON. (Vol. XV., 692).—Dinitro-oxy-anthraquinone is suspended in boiling water and a small quantity of concentrated caustic soda solution is added. A dark red sodium salt is formed. The solution is concentrated until a flocculent precipitate begins to fall. This is filtered, washed with alkali, and treated with hydrochloric acid. The yellow precipitate is crystallized from glacial acetic acid. The analysis shows the product to be mono-nitro-alizarin. The reaction takes place in correspondence with the following equation :



A portion of the nitro group escapes in the form of ammonia.

**On the Reducing Properties of Living Protoplasma.** O. LOEWAND, Th. BOKARNY. Vol. XV. p. 695. Controversial.

**Synthesis of Para-propyl Benzoic Acid.** RICHARD MEYER and ERWIN MUELLER. Vol. XV., p. 698. Para-normal propylbenzoic acid was obtained by the action of moist carbonic acid on brom-propyl-benzol and sodium. The analysis suggests the formula  $C_{10}H_{12}O_2$ .

**Preparation of Lactic Acid.** HEINRICH KILIANI. Vol. XV., p. 699. The results of experiments are : 1. The best crude material is invert sugar ; 2. Caustic soda is best ; 3. It is not advantageous to neutralize the entire alcoholic solution with carbonate of zinc. A detailed method of manipulation is appended.

**Contribution to the Knowledge of Saccharin.** H. KILIANI. Vol. XV., p. 701. Calcium hydrate acts on invert sugar and forms lactic acid. Saccharin was produced as by product. A treatment with oxide of silver shows saccharin to differ in its reactions with this reagent from dextrose and levulose. These, as the author has previously shown, when oxidized in this manner, produce glycolic acid only, besides carbonic, oxalic and traces of formic

acids, while from saccharin acetic acid is produced in addition to this acid. This result seems to point out that saccharin contains a methyl group moreover, because when oxidized with potassium permanganate, not only carbonic acid and water, but also acetic acid are formed.

**On the Molecular Structure of the Metals.** S. KALISCHER. Vol. XV., p. 702. An elaborate paper, giving the results of experiments with the most common metals. The results of all the enumerated experiments may be gathered thus: that the crystalline structure of most metals is their natural condition, and that this may be taken from them, with more or less difficulty (from some not at all), by mechanical effects, and that many of them can be reconverted into this condition by the aid of heat. By assuming this, the cause of the great electrical conducting power of metallic wires, which they assume after being heated, can be explained.

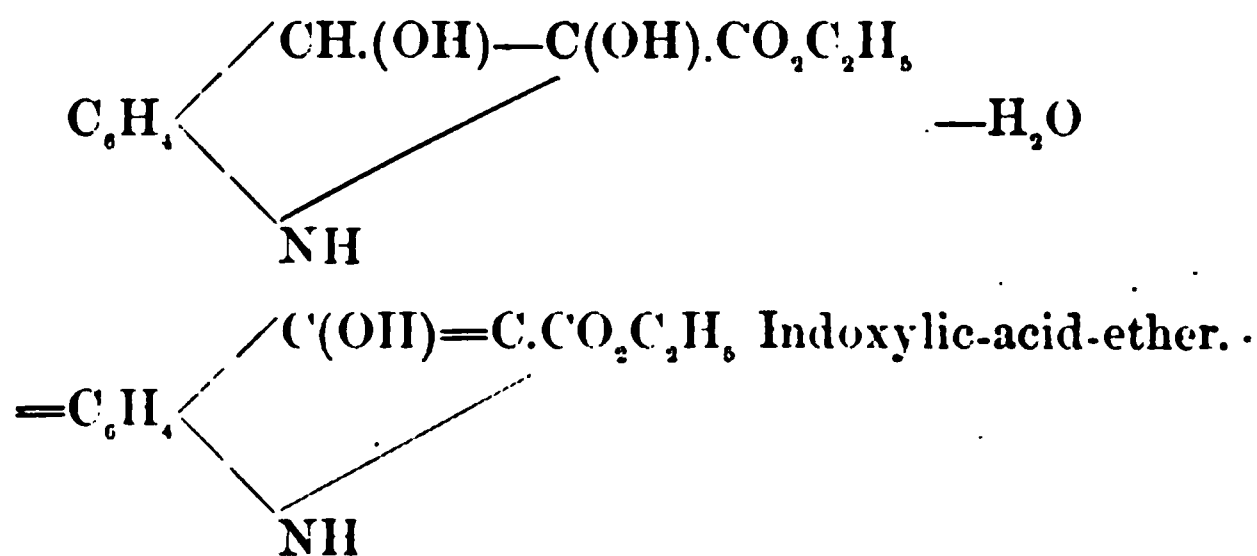
**On Isocholic Acid.** P. LATSCHINOFF. Vol. XV., p. 713. Partly controversial. The author gives various points showing the difference between cholic and isocholic acids.

**On the Action of Bromine in Alkaline Solutions on the Amides.** A. W. HOFMANN. Vol. XIV., p. 2725. Vol. XV., pp. 407, 752, 762.

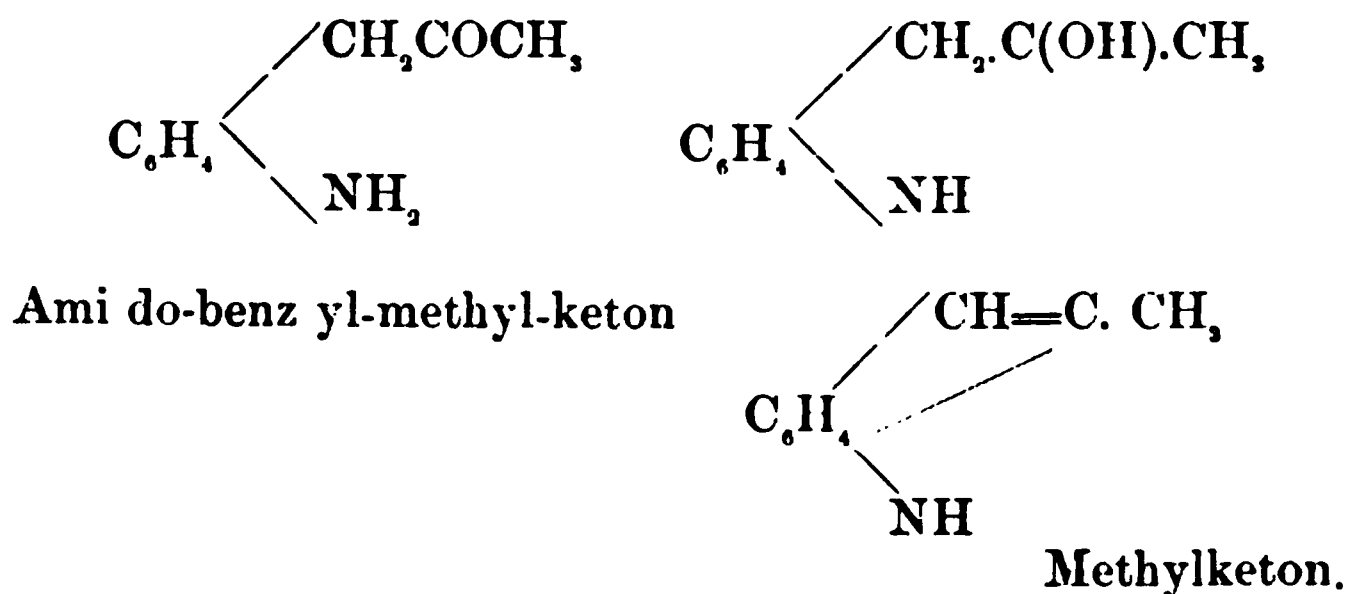
**On the Compounds of the Indigo Group.** ADOLPH BÆYER. Vol. XV., p. 775. By oxydation of indoxylic-acid-ether by means of acid oxydizing agents three consecutive stages are observed:

Indoxylic-acid-ether .....	$C_{11}H_{11}NO_3$
Indoxanhydic-acid-ether .....	$C_{22}H_{20}N_2O_6$
Indoxanthinic-acid-ether .....	$C_{11}H_{11}NO_4$
Ethyl-oxalyl-anthranilic acid .....	$C_{11}H_{11}NO_5$

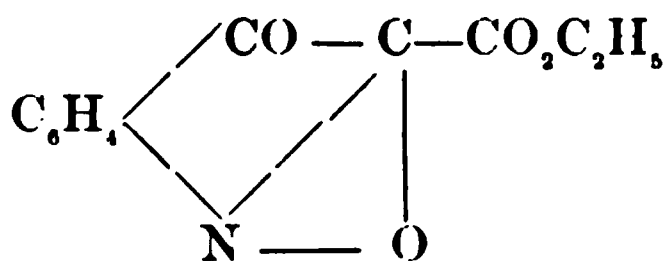
Reduction of indoxanthinic-acid-ether takes place in two stages when heated with zinc-dust and acetic acid.



corresponding to the formation of methyl keton from amido-benzyl-methyl-keton.



The constitution given to isatogenic-acid-ether is shown by the formula.



Hence a group  $\text{N}-\text{C}$  is taken up. The author terms this the “carbazoxy group.” The action of nitrous acid on the various indoxyl compounds is also discussed by the author. The relations of the indol to the to the indoxyl group are as yet uncertain.

**On Monochlorocinamic Acid.** G. W. JUTZ. Vol. XV., p. 788. By proper treatment of phenyl-dichlor-propionic acid with potassium tartrate, true potassium-chloro-cinamates are formed. They are separated by difference of solubility in alcohol. From the salts two acids were obtained. Monochloro-cinamic-acid melting at  $142^\circ$ – $143^\circ$ , and chloro-cinamic-acid at melting at  $114^\circ$ .

**Investigations on the Diffusion of some Organic and Inorganic Compounds.** J. D. R. SCHEFFER, Vol. XV., p. 788.

**On Di- and Tri-Phenylphosphin.** A. MICHAELIS and L. GLEICHMANN. Vol. XV., p. 801. Diphenyl-phosphin is formed by treating diphenyl phosphin chloride  $(\text{C}_6\text{H}_5)_2\text{PCl}$  with water, or dilute solution of caustic soda, diphenyl-phosphinic acid or its sodium salt being formed at the same time :

$2 (\text{C}_6\text{H}_5)_2 \text{PCl} + 2 \text{H}_2\text{O} = (\text{C}_6\text{H}_5)_2 \text{PH} + (\text{C}_6\text{H}_5)_2 \text{PO.OH} + 2 \text{HCl}$ . Triphenyl-phosphin  $(\text{C}_6\text{H}_5)_3\text{P}$  is formed when a solution of phosphenylic chloride and brombenzol is left in 3 to 4 times the volume of ether (free from water) with thin strips of sodium. This left to itself for 6 or 10 days at the common temperature,  $\text{C}_6\text{H}_5\text{PCl}_2 + 2\text{C}_6\text{H}_5\text{Br} + \text{Na} = (\text{C}_6\text{H}_5)_3\text{P} + 2\text{NaCl} + \text{Na Br}$ .

**On Beta-Naphtol-Aldehyd.** G. KAUFFMANN. Vol XV., p. 804. 40 grms. of beta-naphtol are dissolved with 60 grms. of caustic soda in 250 grms. of water and heated on a water bath with 50–60 grms. of chloroform at an upright condenser for several hours. Three compounds are formed. Petroleum benzene dissolves out the aldehyde and a second compound. These two may be separated by acid of caustic soda solution in which this second substance is insoluble. The aldehyde is purified by driving off with steam. The experimenter then formed an acid in order to characterize the aldehyde. A di-naphtol is also formed.

**Note on artificially colored claret wines.** P. PASTROVICH, (Vol. XV, p 808.)

Presence of fuchsin is detected by treatment of the wine with oxide of manganese (braunstein). The wine is not deprived of its color when colored with fuchsin and treated in the above manner 2 mgs. of fuchsin to the litre can be detected.

**On the bases produced from the products of addition of chinolin and halogenalkyls.** W. LA CASTE, (Vol. XV, p 809.) Controversial.

**On the action of lead and manganese dioxides on halogen metals in presence of acetic acid.** C. L. MUELLER and G. KIRCHNER, (Vol. XV, p 812.) Controversial.

The chlorine liberated from hydrochloric acid by means of the binoxide of lead seems to convert the acetic acid into monochloro-acetic acid and this again by the formation of lead chloride, carbonic acid and free chlorine is farther oxidized. An aqueous solution heated with  $\text{Pb O}_2$  produces the products enumerated and after continued boiling of  $\text{Na Cl Pb O}_2$  and acetic acid the liquid distilled contains a considerable quantity of monochloro-acetic acid. The process is different when binoxide of manganese is used. No chlorine, but only carbonic acid is generated. The monochloro-acetic acid is oxidized to carbonic acid and water and manganous chloride is formed.

**The nomenclature of complex azo-compounds.** K. HEUMANN, (Vol. XV, p 813.)

The author proposes to name azo-bodies in which two azo-groups are bound to the same hydrocarbon rest diazo compounds. In order to form the name the term—disazo—is appended to the name of the hydrocarbon rest carrying the two azo rests and to this the rests connected with the azo groups are attached. For example: Resorcin-disazo-benzol, benzol-disazo-benzolresorcin.

**On the occurrence of Carvacrol in the essential Oil of Satureja Hortensis.** E. JAHNS, (Vol. XV, p 816.)

The analysis in round numbers of the oil examined:

30% carvacrol.

20% cymol.

50% of a terpen (boiling between 178–180°) besides traces of a phenol having the property of turning iron blue.

**On Tetrabromchinolin and Diiodochinolin.** AD. CLAUS and E. ISTEI, (Vol. XV, p 820.)

By the action of the halogens in bisulphide of carbon solution two new products were obtained. When chinolin is dissolved in bisulphide of carbon and a solution of bromine in a considerable quantity of bisulphide is gradually added and the solution is then at once evaporated, a red-brown mass is obtained, which analysis shows to be tetrabromchinolin. By the action of sodium amalgam in alcoholic solution a portion of the bromine is taken away even in the cold and a new compound containing bromine but of a basic character is formed. This is undoubtedly not a product of addition. A bisulphide of carbon solution of chinolin when treated with iodine. The product is one of addition corresponding to the formula  $C_9H_7I_2N$ .

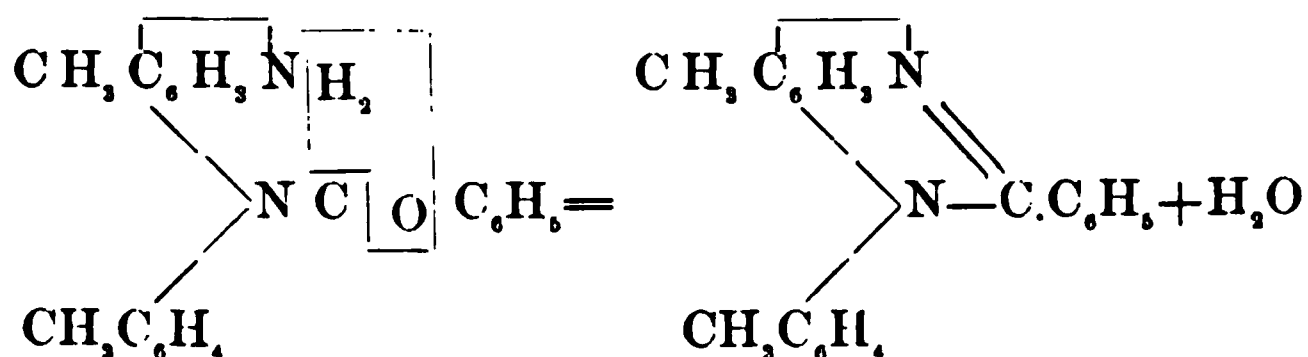
**Contribution to the knowledge of Diphenylamin and Para-ditolyl-amin.** EUG. LELLMANN, (Vol. XV, p 825).

The benzoyl derivative of diphenylamin  $C_6H_5CON(C_6H_5)_2$ , according to the strength of nitrification produces one mono compound benzoyl-para-mono-nitro-diphenylamin and two dinitro compounds benzoyl-ortho-dinitro-diphenylamin and benzoyl-para-dinitro-diphenylamin. By nitrification of benzoyl-para-ditolyl-amin only orthonitro compounds are formed.

**On a New Class of Amidines.** E. LELLMAN, (Vol. XV, p 832.)

The benzol compound of para-ditolylamin was subjected to nitri-

fication and the compound formed, *i. e.* benzoyl-ortho-nitro-ditolylamin was reduced by nascent hydrogen. The benzoyl-ortho-amido-para-ditolylamin by loss of water was converted into the amidin which must be termed :



para-tolyl-benzenyl-toluylen-amidin.

### The Properties and the Atomic Weight of Uranium.

CLEMENS ZIMMERMANN, (Vol. XV, p 847).

Prepared by reducing a mixture of potassium or sodium with chloride of uranium by heating in charcoal crucible. Uranium has the color and lustre of silver but is much harder. Striking with a hammer produces sparks. It oxidizes gradually when exposed to the air. It burns when heated on a platinum foil. It is decomposed by nitric acid. Sp. gr. is 18.7. Author gives the atomic weight equal to 240 making it the greatest of all known metals.

### Contributions to the Knowledge of Anthramin. C.

LIEBERMANN and A. BALLERT, (Vol. XV, p 852).

Prepared from anthral by means of ammonia in aqueous solution Heated in sealed tube with 60 times its volume of ten per cent aqueous ammonia for several hours at 250°. Anthramin can also be prepared by heating for eight hours at the return condenser with acetamid.

### On Chinolin Derivatives. TD. SKRAUP, (Vol. XV, p 893.)

By treating m—nitro and amidotoluol, glycerin and sulphuric acid m—toluchinolin is produced. Partly controversial.

### On the Preparation of the Amids of Monobasic Acids of the Fatty Series. A. W. HOFMANN, (Vol. XV, p 977.)

The dry ammonia salts of the acids are subjected to a temperature of 230° under pressure for five or six hours. The tubes then contain an aqueous solution of the ammonia salt and a large quantity of the amid, which in the most advantageous case varies between 80 and 85% of the theoretical result.

**On the Preparation of Mustard Oils.** A. W. HOFMANN, (Vol. XV. p. 985). The double substituted sulpho-urates of the aromatic amines still serve as a starting point. By abstracting one mol. of monamin the mustard oils are produced. Diphenyl-sulpho-urate produces anilin and phenyl mustard oil.



The author found that concentrated solution of phosphoric acid performs the separation with ease and certainty. In all cases over 90% of the theoretical results were obtained.

**On the Action of Bromine on Bisulphide of Carbon.**—CARL HELL and FR. URECH, (Vol. XV. p. 987).

The result of numerous elaborate experiments is given in brief by the authors as follows: that without doubt bisulphide of carbon is not at all indifferent towards bromine, that excluding the presence of other bodies it forms a product of addition, but that in presence of water, or what is even more important, in presence of organic acids it can be completely oxidized.

**On the Vapor Density of Bromine**—HANS TAHN, (Vol. XV. p. 1238).

Two data only have been given before. Mitscherlich finds the vapor density equal to 5.54 and V. and C. Meyer find it 5.38 at a temperature of 99.34° C. The authors experiments are tabulated. If the vapor densities found at different temperatures are collected in a formula

$$D = a + bt,$$

then by the aid of the method of the smallest squares of the constants  $a$  and  $b$  the probable values obtained are

$$a = 5.8691 \text{ and } b = 0.00153$$

The conclusive table given is (for various temperatures) as follows:

Temperature above boiling point	Density	Deviation from the normal	Difference.
40°	5.7115	3.381	0.554
60°	5.6809	2.827	0.554
80°	5.6503	2.273	0.554
100°	5.6197	1.719	0.554
120°	5.5891	1.165	0.553
140°	5.5585	0.612	0.554
160°	5.5279	0.058	



**On the Expansion of Alums.** W. SPRING, (Vol. XV. p. 1254). The alums expand equally between  $0^{\circ}$  and  $60^{\circ}$ . This simple result which from all appearances is due to the isomorphism of the compounds finds its confirmation in the fact that large potassium, or ammonium-alum crystals can be grown on a chrom-alum core. It is found from the results of the investigation that *Avogadro's* law can be extended to solid bodies and hence the question of determining the molecular weight of solid bodies may soon be defined from this.

**Quantitative Determination of Fusel Oil in Brandy.** L. MARQUARDT, (Vol. XV. p. 1370).

In 1000 grms. of a 30% brandy, prepared by mixing of 95% alcohol with water, one gram. of commercial amylic alcohol was dissolved, of this 150 grms. were shaken for 15 minutes with the same volume of water and 50cc of chloroform, and this operation repeated twice with 50cc more. The united 150cc of chloroform are united and again shaken up for 15 minutes with the same volume of water and this is repeated twice with the same quantity of water. The chloroform now containing all the fusel oil and no more alcohol was heated in a well stoppered bottle in a water bath at  $85^{\circ}$  for about 6 hours together with a solution of 5 grms. of bichromate of potash in 30 grms. of water and with 2 grms. of sulphuric acid. After completed oxidation contents of flask including the chloroform were put into a distilling flask the residue washed out with water and the whole distilled off to about 20cc. The residue was covered with 80cc of water and distilled off, leaving about 5cc. The distillate, consisting of two layers, was mixed with barium carbonate and digested in the return condenser for 30 minutes. Chloroform was then distilled off, and the remainder evaporated down to about 5cc. The excess of barium carbonate was filtered off, washed with very little water and the filtrate evaporated to dryness. The remainder which weighed 0.396 grms. was dissolved in 100cc of water and a few drops of nitric acid. 50cc were employed for the baryta determination and 50cc for the barium chloride determination. The chlorine determination for 100cc gave 0.0638 grms. of barium chloride which is to be subtracted from 0.396 grms. of the above residue. The difference is 0.3322 grms. 0.18004 grms. of baryta were obtained from 100cc. The baryta corresponding to the chloride must be subtracted *i. e.* 0.0469 grms. There remains 0.13314 grms which is contained in 0.3322 grms. of baryta

salt and hence constitutes 40.07% of it. As barium valerianate contains 45.13% of baryta, this salt is the greater constituent of the baryta salt. For one equivalent of baryta two molecules of anylic alcohol are calculated. In the foregoing case 0.13314 grms. of baryta give 0.15315 grms. of anylic alcohol or fusel oil in 150 grms. or 102 grms. in 1000 grms. of brandy.

**Artificial Piperin.** L. RUEGHEIMER (Vol. XV. p. 1390). When the chloride of piperinic acid is treated with excess of piperidin (both being in solution in benzol freed from water) chloride of piperidin is separated. After warming and filtering off the chloride, the benzol solution is shaken with dilute hydrochloric acid. The solution is allowed to stand and piperinic acid separates. This is filtered off, the greater part of the benzol is distilled off, ligroin (petroleum naphtha) is added until a permanent precipitate is formed. The solution is then allowed to evaporate off gradually and piperin remains in form of well developed crystals.

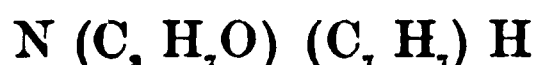
**On the Alterations of Preserved Milk.** O. LOEW (Vol. XV. p. 1482). A sample of milk of 400cc. was heated 40 minutes to 101° and examined eight years after. The milk sugar had disappeared and had been transformed into its hydrolytic products of division (lactose and glyucose). The casein and albumen had been transformed into pepton. A portion of the pepton had undergone another change, for leucin tyrosin and ammonia were found. The tyrosin was most probably present as its anhydride.

**On a New Class of Dye Stuffs.** O. FISCHER and CH. RUDOLPH (Vol. XV. p. 1500). The name flavanilin is proposed. The product of heating together for several hours acetanilid and chloride of zinc to 250°—270°, is boiled out with hydrochloric acid, and the dye is precipitated out of the solution by salt and a small addition of acetate of soda. The color of the dye is a beautiful yellow. The ethyl compound is obtained by heating flavanilin with ethyliodide under pressure. The color is orange on silks. Flavenol is the nitrous compound. Flavolin is obtained from flavenol by mixing with zinc-dust and distilling at a dull red heat.

Abstracts from the *Journal of the Chemical Society*, London, by Arthur H. Elliott,  
Ph. B., F. C. S.

**On Oxypropyltoluidine.** By H. FORSTER MORLEY, M. A.  
(Vol. XLI. p. 387.)

This body is prepared by dissolving paratoluidine in propylene oxide, and distilling. It comes over between 285°—296° C. and is recrystallized from benzol. Its formula was found to be



Another method of preparing this body is by allowing the above mixture to stand some days, when the new base crystallizes out, and can be re-crystallized from petroleum oil. A third method is by converting the bases into oxalates and crystallizing, the oxalate of the new base being soluble.

Oxypropyltoluidine melts at 74° C, boils at 293° C, is insoluble in water, but soluble in benzol, ether, alcohol, and petroleum. It forms an oxalate with the composition  $\text{C}_{10}\text{H}_{11}\text{NO}, \text{H}_2\text{C}_2\text{O}_4$ .

The author also describes the result of distilling oxypropyltrimethylammonium hydrate, the chief product being propylene glycol, with trimethylamine and carbonic acid; the glycol being mixed with small quantities of other bases.

**On some Halogen Compounds of Acetylene.** By R. T. PLIMPTON, Ph. D. (Vol. XLI. p. 391).

Author prepared the tetrabromide, boiling at 110°–111°C, and having a specific gravity of 2.268 at 0°C. The di-iodide was prepared by passing acetylene over iodine moistened with alcohol; it melts at 73°C; it boils at 192°C and is distilled without decomposition. The chloriodide  $\text{C}_2\text{H}_2\text{HCl}$  was prepared by passing acetylene through iodine monochloride. It boils at 119°C and the specific gravity at 0°C is 2.2298. The chlorobromide was obtained by treating the chloriodide with bromine; it boils at 81°–82°C Sp. gr. at 0°C=1.8157. Acetylene Bromiodide is prepared by passing acetylene through bromine iodide; its Sp. gr. at 0°C (solid) is 2.750 and at 17°5C=2.6272; it boils at 150°C.

**On Dihydroxybenzoic Acids and Iodo-Salicylic acids.** By ALEX. K. MILLER, Ph.D. (Vol. XLI. p. 398).

Author prepared dihydroxybenzoic acids from catechol by heating in a sealed tube with ammoniac carbonate, at 130°–140°C for 14–16 hours. Two acids were obtained, protocatechinic acid, and

an acid with the formula  $C_7H_6O_4$ , which differed from the former acid in its crystalline form and its reactions with ferric chloride.

The author also prepared this acid by the action of iodosalicylic acids, obtained by the direct action of iodine upon salicylic acid, and melting potash. The barium salts of the mono and di-iodosalicylic acids were prepared, but could not be separated. Finally the two acids themselves were separated by fractional crystallization. Two crops of crystals were obtained, one melting at  $197^\circ$  and the other at  $198^\circ C$ . The former is para-iodosalicylic acid and analysis gave the formula  $C_6H_4I(OH).COOH$ . This acid was heated with potash and treated with excess of hydrochloric acid. The liberated dihydroxybenzoic acid was separated by ether, and crystallized from water. Its solution gives no precipitate with lead acetate. It melts at  $200^\circ C$ . Heated to  $215^\circ C$  it gave pure quinol. Analysis gave the formula  $C_6H_4(OH)_2, COOH$ .

The second more soluble iodosalicylic acid obtained above by crystallization melts at  $198^\circ C$ . Analysis gave the formula  $C_6H_4I(OH).COOH$ . By melting this body with potash a dihydroxybenzoic acid was obtained, which differed from the above dihydroxybenzoic acid in its crystalline appearance and reactions with ferric chloride. It also gives a precipitate with lead acetate; and on heating yields carbonic acid and catechol. It melts at  $204^\circ C$ . Analysis gave the formula  $C_7H_6O_4$ . The acid so obtained is identical with that obtained from catechol at the beginning of the paper. This completes the series of the six isomeric dihydroxybenzoic acids, and the author gives a valuable table showing the properties of each.

# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

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*Regular Meeting, February 2, 1883.*

Mr. James H. Stebbins, Jr., in chair.

The minutes of November, December and January meetings were read and approved.

The following gentlemen were unanimously elected: Messrs. Percy Neymann, J. Howard Wainwright, Max Schwartz, Vincent M. Picabia, Jas. L. Howe, Andrew Peters, F. T. Walsh and T. Lambert.

Mr. Chas. Eimer (130 E. 18th St.) was then nominated by James H. Stebbins, Jr., Wm. Rupp, and Aug. Eimer.

Remarks upon the last paper were then made by Messrs. Waller, Casamajor and Gladding.

Mr. Gladding explained at length his plan of sampling soap and determining water, as follows :

To prepare an average sample, one inch in length is taken from the middle of the bar and is thoroughly mixed with a stout spatula.

For the determination of the water, a small beaker (100 cc. capacity) is accurately weighed. Into this is then weighed a half inch in depth of pure dry quartz sand, and also a small glass rod. About 5 grams of the thoroughly mixed sample is then placed in the beaker and accurately weighed. 25 cc. of 95 per cent. alcohol are then poured into the beaker and the whole placed in a water oven. The alcohol dissolves the soap, and the contents of the beaker are stirred at intervals by means of the glass rod. As the alcohol evaporates, the soap is left in a very thin coat upon the particles of sand and the expulsion of water is very complete. The perpendicular sides of the beaker prevent any loss of alcohol from creeping.

Mr. Gladding suggested that the residue might then be treated with hot petroleum ether, the ether decanted and the treatment repeated several times and the residue again dried. The loss would be uncombined fat. The residue might then be dissolved in 95 per cent. alcohol, the solution filtered and the filtrate and residue carried through the several stages proposed by Dr. Leeds.

As he had had trouble determining the uncombined alkali by the titration recommended by Dr. Leeds, the precipitation of this by a

current of  $\text{CO}_2$ , followed by filtration and estimation of the precipitated carbonates, might be found more reliable.

The following papers were read :

1. "On the Action of Diazoanisoie Chloride upon Phenoles and their Substitution Products," by Jas. H. Stebbins, Jr.
2. "On Acrolein Urea, with remarks upon Prof. Hugo Schiff's publications upon Condensed Ureas," by Dr. A. R. Leeds.
3. "Detection of Anhydrous Glucose mixed with Refined Cane Sugar," by P. Casamajor.
4. "A new Scheme of Soap Analysis, with a preliminary discussion of former methods," by Dr. A. R. Leeds.

The resignation of Mr. M. Benjamin was accepted.

The meeting then adjourned.

THOS. S. GLADDING,  
Recording Secretary.

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## ACTION OF DIAZOANISOLE CHLORIDE UPON PHEN- OLES, AND THEIR SUBSTITUTION PRODUCTS.

BY JAMES H. STEBBINS, JR.

On the 29th day of Jan., 1879, Peter Griess filed a specification for the production of a red coloring matter, obtained by the action of diazoanisoie chloride upon an alkaline solution of the beta sulpho acid of beta naphthole.

On Feb. 12, 1879, he filed another specification, for the production of a crimson dyestuff, formed by the action of diazoanisoie chloride upon an alkaline solution of the disulpho acid of beta naphthole.

These two specifications, therefore, teach us that the diazo-ethers of phenoles can react upon phenoles, as well as the more ordinary diazo-compounds.

Since the dyestuffs produced in the above manner are but few in number, I thought it worth my while to complete, if possible, the series already so well begun.

For this purpose the following experiments were made :

### *Orcine-azo-benzole-di-soda salt.*

12.3 grms. anisidine were dissolved in 17.9 grms.  $\text{HCl}$ . (1.20 sp. gr.), diluted with 200 cc. water.

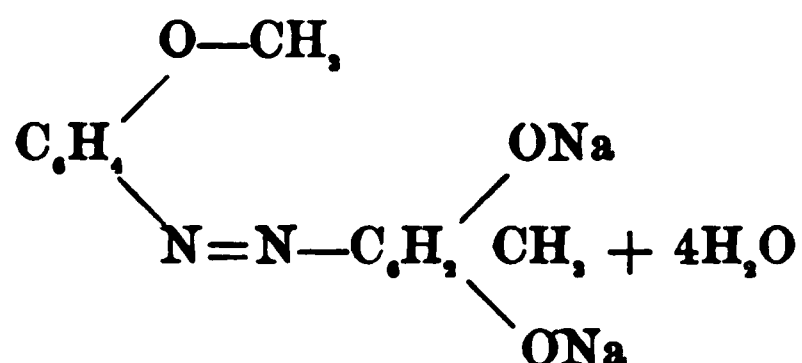
The solution thus obtained was diazotized with 6.9 grms.  $\text{NaNO}_2$ , in 100 cc.  $\text{H}_2\text{O}$ , and allowed to rest for about 1 hour.

It was then added, under constant stirring, to a solution composed of 12.4 grms. orcine, 200 cc. water; 8 grms.  $\text{NaOH}$ , and 10 cc.  $\text{NH}_4\text{OH}$  and A q.

The orange colored precipitate thus formed was collected on a filter, washed repeatedly with cold water, to remove all  $\text{NaCl}$ , and finally dissolved in boiling water.

The solution on cooling deposits the soda salt, in the shape of little yellow-brown needles, having in reflected light a beetle-green color.

This body probably has the following formula :

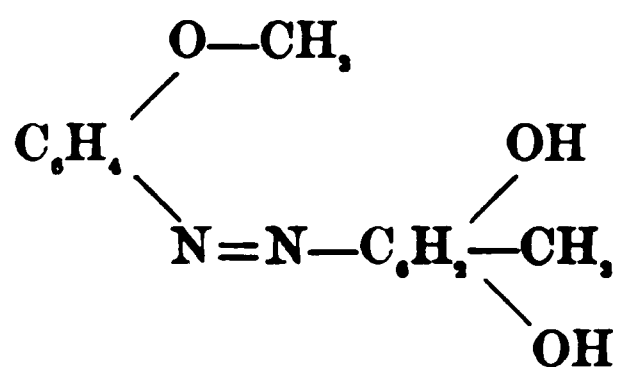


I say probably, because the figures derived from several analyses do not agree with one another, or with theory.

The above substance is insoluble in cold, and sparingly soluble in boiling water, from which it crystallizes with four molecules of water of crystallization.

It is readily soluble in alcohol, but cannot be obtained therefrom in a crystalline state.

*Orcine-azo-anisole.*



This substance was obtained from the soda salt, by dissolving the latter in  $\text{H}_2\text{O}$ , and adding strong  $\text{HCl}$  in excess.

A heavy brick red precipitate is thus formed, which when examined under the microscope, is found to be composed of very minute, hair fine orange-colored needles.

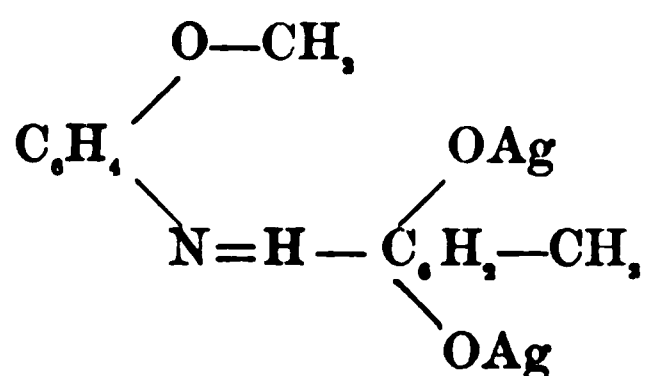
It was collected on a filter, washed with boiling water, till free from all  $\text{HCl}$ , and dried in the air bath at  $120^\circ \text{C}$ .

A combustion of the substance thus prepared, gave the following figures :

<i>Found.</i>	
Carbon .....	65.95 per cent.
Hydrogen .....	6.16   “
<i>Theory.</i>	
Carbon .....	65.11.   “
Hydrogen .....	52.4   “

When heated on platinum foil, it swells up, giving off yellow fumes, and leaving as residue a large amount of porous carbon.

*Orcine-azo-anisole di silver salt.*



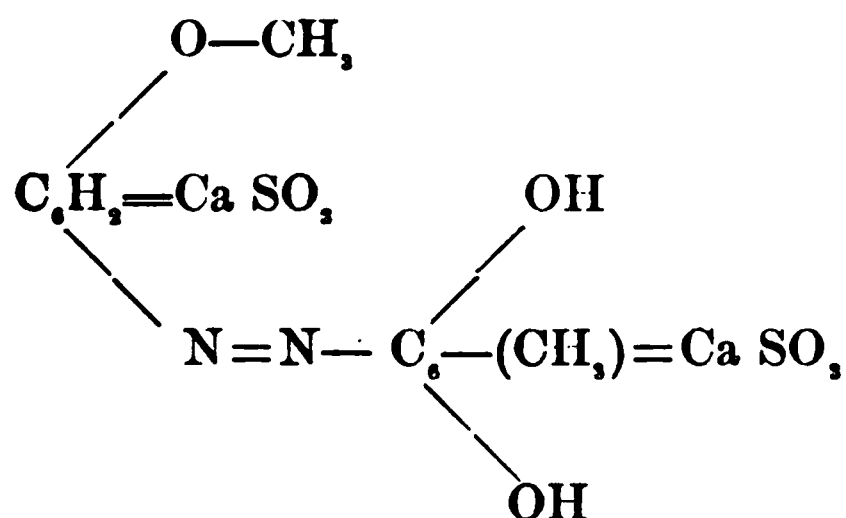
This body was obtained from a hot aqueous solution of the soda salt, by adding  $\text{AgNO}_3$  in excess. The blood red precipitate thus formed, was collected on a filter, and washed with boiling water, till free from all traces of  $\text{AgNO}_3$ , and finally dried at  $110^\circ\text{C}$ . It is insoluble in water, and but sparingly soluble in alcohol.

Owing to its properties of not crystallizing, it was impossible of obtaining a fair sample for analysis, and therefore the figures obtained are somewhat too high.

0.1047 grms. silver salt gave 0.0530 grs. Ag.  
 Equivalent to Ag.                      50.62%

<i>Theory.</i>	
Silver .....	45.76%

*Orcine-azo-anisole-disulphonate of lime.*





This interesting substance was obtained by heating 1 pt. of orcinè-azo-anisole with 3 pts. of very strong fuming sulphuric acid, on the water bath for about 1 hour.

The beetle green melt thus obtained, was powdered into hot water, and neutralized with carbonate of lime. The  $\text{CaSO}_4$  was collected on a filter, and the filtrate containing the lime salt, evaporated to dryness.

This salt is a yellow-brown, slightly crystalline powder, and is readily soluble in water with an orange color.

A calcium estimation gave the following figures :

*Found.*

Calcium.....15.29%

*Theory.*

Calcium.....16.06%

Furthermore a sulphur estimation gave :

Sulphur.....13.71%

*Theory.*

Sulphur.....12.85%

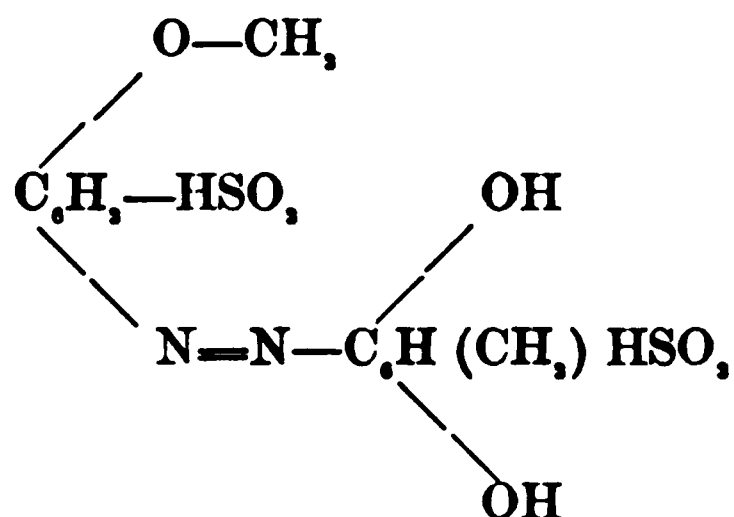
*Reduction Products.*

The above orcinè-azo-anisole disulphonate of lime was dissolved in water, and boiled with an excess of stannous chloride, until the solution became nearly colorless.

The tin was precipitated with caustic soda, and collected on a filter. The filtrate was then shaken up with ether, hoping thereby to take up the reduction products.

After the ether had been evaporated, a small amount of a white crystalline body remained behind, but no oil drops of anisidine were to be found.

It may therefore be surmised that the original sulpho acid, has the following constitution :



In other words, that the sulpho groups are evenly distributed



memoir, but an incorrect extract therefrom. I wish to state expressly that this was not the case, and that my own experiments were conducted with constant reference to Mr. Schiff's original memoir in the Ann. Chem. Pharm. loc. cit., and that I did not isolate the compound containing the residue from 2 molecules of urea, but only the compound with the residue from one molecule or  $\text{CO. (NH), C, H}_4$ . Furthermore, I wish to state that the non-crystalline, white body which I obtained by the purification of the precipitate which ensues when acrolein is added to an alcoholic solution of urea, and found to be  $\text{CO. (NH), C, H}_4$ , is the same compound as is obtained when an aqueous solution of urea is treated with acrolein, in the manner described by Mr. Schiff, but which he did not purify adequately, and to which he consequently gave the erroneous formula of an Acryldiureid.

In his criticism upon my article, Mr. Schiff says that he analyzed this compound which he obtained with an aqueous solution of urea, and that it contained 38.3 per cent. carbon, the right percentage for Acryldiureid. But in the memoir to which Mr. Schiff has referred me no such analysis is given, and if this analysis is not contained in that memoir, I desire to be referred to some article published before this reply of Mr. Schiff, in which it can be found.

I have stated that I was able to isolate, and that with much difficulty, but one compound in a state of purity, and for that reason I analyzed only this one compound, finding in it Carbon 49.01 per cent. (accidentally misprinted in the Berichte 40.01 per cent.) and Hydrogen 6.30 per cent. There is undoubtedly a resinous, oily by-product, to whose presence the difficulty of obtaining the pure compound is due. But Mr. Schiff has analyzed the various mixtures obtained after more or less partial purification, and deemed that he was entitled on the strength of such analyses so conclude in the first place that he had *isolated* Acryldiureid, and that another body, which he did not isolate but obtained as a white porcelain-like substance intimately mixed with acrolein was Diacryltriureid, 42.2 per cent. Carbon and 6.3 per cent. Hydrogen. The figures which he obtained by analysis of this mixture, and which are the only figures given in the memoir he has referred me to, varied between C. 44–47 per cent. and H. 6.9–7.1 per cent.

The method which Mr. Schiff employed to obtain his Diacryltriureid, by treating pulverized urea directly with acrolein, no solvents being employed, was one which required more than usual care and perseverance in the consequent treatment of the product with

solvents in order to separate the bodies thus formed in a state of purity. No such precautions are stated to have been used. On the contrary in his reply to my article, Mr. Schiff has persisted in his plan of making analyses of mixtures, and from his results, he has inferred the presence in these mixtures of no less than three compounds, the composition of anyone of which differs widely from the figures actually obtained.

I think the majority of chemists would find it difficult to believe that Mr. Schiff has endeavored to support the erroneous conclusions arrived at in his original memoir in the *Ann. Chem. Pharm.*, by methods even more inexact and reprehensible, were they not to read his own statements contained in the *Berichte* XV, 1395. He states there that a portion of the product obtained by the direct action of Acrolein upon Urea, after keeping for 14 years, had undergone a peculiar alteration. About two-thirds had become of a strong canary color, "und man sieht deutlich, wie die Stücken von einer weissen Masse durchzogen sind, so dass die gelben Stücken ofters centrale weisse Stellen zeigen und hierdurch oxalartiag und fast perlmuttähnlich aussehen. Die Antheile von Krystallinische Aussehen haben dieses Aussehen sowie ihre weisse Farbe bewahrt, aber man Kann jetzt auch bei diesen Stückchen, bei Vergrosserung, um so besser sehen, dass noch gelbe und porcellanartig weisse Substanz innigst beigemengt ist. Leider war gerade diese letztere, weniger cohärente Masse grössten theils zu grobern Pulver zerfallen."

And yet the constituents of a mixture like this, which it would have required great care to have properly separated by means of appropriate solvents, Mr. Schiff attempted to separate by mechanical means, and to pick them out by the aid of a magnifier.

He thus obtained (1) "Gelbe amorphe Substanz mit wenig wiesser porcellanartiger Substanz durchzogen," and with the composition C. 46.81 p ct., H. 7.09 p ct; (2) "Weisse, auscheineud Krystallinische Substanz, noch etwas amorphe weisse und gelbe Verbindung einschliessend," with the composition, C. 45. 15 p ct., H. 6.54 p ct. From these anylyses by a very complicated process of inductive logic, Mr. Schiff concludes that his mixture analyzed consisted of no less than three different compounds, viz :—Acrylureid C. 49 p ct., and H. 6.1 p ct; Acryldiureid C. 38 p ct., and H. 6.3 p ct., together with Acrylharz C. 60.61 p ct., and H 7-7. 5 p ct.

When we remember that in his original memoir Mr. Schiff stated that the mixture above analyzed contained Diacryltriureid C. 42.2

p ct., and H. 6.3 p ct., and now are told that by a process of logic he infers from analyses performed upon the same material, but much altered after standing 14 years, he has shown that the mixture was really Acrylureid, Acryldiureid and Acrylharz, we are in a position to know how much reliance should be placed either upon the work as first performed, or as afterwards corrected. Mr. Schiff has never isolated Acrylureid, but accepts my own statement as proof of its existence; he has never isolated Acryldiureid, but on the contrary has called Acryldiureid what was in fact impure Acrylureid, and yet in this final statement he affirms that both these bodies exist in a certain mixture together with an unknown quantity of a resin.

ÆNANTHUREIDE, BENZUREIDE, ÆNANTHO-BENZUREIDE, ETC.

But the foregoing are the smallest among the many marvellous results which Mr. Schiff has arrived at, by applying a great deal of logic to the analyses of complex mixtures. In the original memoir to which he has referred me, he states that he obtained by treating a strong alcoholic solution of Urea with alcohol, Ænanthodiureid,  $C_6 H_{12} N_4 O_8$ , (analysis not given) as a crystalline body melting at  $166^\circ$  to a yellow oil. Further on that he obtained by treatment of Ænanthol with crystals of Urea, Diönanthotriureid,  $C_{11} H_{22} N_6 O_{11}$ , (analysis not given), as a crystalline body melting at  $162^\circ$  to a yellow oil. When these two Ænanthureide are treated again with small quantities of Ænanthol, water is formed and the ureide change into "zähe hornige" masses. After cooling these masses are pulverized as far as possible, and extracted with ether. In this manner condensed Ureas are obtained, Trioenanthotetrureid,  $C_{22} H_{44} N_{12} O_{22}$ , and Pentoenanthohexureid,  $C_{31} H_{62} N_{16} O_{31}$ . The former a yellow powder melting about  $155^\circ$ , the latter is a heavy mass which appears to melt at  $150^\circ$ .

When the raw product obtained in the preparation of this hexureid is treated with alcoholic ether and the extract evaporated, there remains behind an amorphous residue. Acetone takes out some ænanthol and leaves a gelatinuous mass which dissolves in alcohol. The solution at a certain degree of condensation solidifies to a glue like substance, which dries to a brittle varnish. This body consists of *twelve* molecules of urea united by the residues from *eleven* molecules of ænanthol, or Dodekoenanthondekaureid:



	<i>Calculated.</i>	<i>Found.</i>
Carbon . . . . .	60.1	60.5
Hydrogen . . . . .	10.1	10.6

But why, according to Schiff's methods of reasoning, should not this glue-like substance solidifying to a varnish be inferred from the analysis to consist of sixteen molecules of urea united with the residues from 15 molecules of œnanthol, this supposition agreeing better with his own results !



	<i>Calculated.</i>	<i>Found.</i>
Carbon . . . . .	60.5	60.5
Hydrogen . . . . .	10.2	10.9

It is an extremely fortunate circumstance that, as Mr. Schiff remarks, the ureas more condensed than the hexureid are so very similar in their constitution and so difficult of purification, that he was unable to establish their formulæ "*with certainty.*"

For he truly says, that these substances are very remarkable in regard to their contained nitrogen; since no continuations have ever yet been artificially produced with so large a number of nitrogen atoms. If more of such compounds had been produced the complex nitrogenous substances of animal origin would have appeared simple in comparison.

It is needless to discuss the Benzo-diœnanthotetrureid  $C_{26} H_{44} N_8 O_4$  and the Benzo-tetrœnanthohexureid  $C_{41} H_{76} N_{12} O_6 = 6 CH_4 N_2 O + 4 C_7 H_{14} O + C_7 H_6 - 5 H_2 O$ . Analyses are given which agree sufficiently well with this formula, but they likewise agree with other formulæ which might quite as reasonably be assigned, and it is a dangerous precedent to assign value to analyses of a body stated to be like *dried fibrine* (!) and of whose purity there is no proof whatsoever.

All of the foregoing, and the further work of Mr. H. Schiff upon Anisureïde, Salicylureïde, Æthylsalicylureïde, Æthylidenharnstoff, etc., etc., must be repeated, and his results verified, before his views upon the constitution of these condensed ureas can be regarded as worthy of acceptance.

## DETECTION OF ANHYDROUS GLUCOSE, MIXED WITH REFINED CANE SUGAR.

BY P. CASAMAJOR.

I lately received from Chicago a sample of sugar, suspected of being adulterated. The appearance of this sugar, and the sensation to the touch, were decidedly in favor of the supposition that starch sugar had been mixed with it. The sensation to the touch is very characteristic of such mixtures. The person who sent the sugar described this sensation by saying that "it felt like ground coconut."

Upon applying to the suspected sugar several tests, which had been found efficacious in detecting common hydrated starch glucose, the following results were obtained :

A test which was described before this Society at its meeting of March, 1880,\* consists in stirring a suspected sugar with a small quantity of water. If any ordinary commercial starch glucose is present, this will appear as chalky white specks, which are distinctly seen in the translucent mass of wet sugar. This test is worthless with crystallized anhydrous glucose, as the crystals of this substance have, when mixed with water into a paste, the same translucency as crystals of cane sugar.

I also applied to the suspected sugar, the test which consists in treating a sample of sugar with a saturated solution of starch glucose in methylic alcohol of density equal to 50° of Gay Lussac's alcohometer.† In the present instance, the saturated glucose solution which I used was made from anhydrous dextrose. By washing the suspected sugar with this solution, a small crystalline residue was obtained, which could not represent the total glucose present, as there was only 5 p. c., a quantity too small to pay for the trouble of mixing it with refined sugar.

The crystalline residue, placed under a microscope, showed the elongated square prisms, characteristic of anhydrous glucose, a form which cannot be mistaken for crystals of cane sugar, and still less for the thin plates of hydrated glucose.

Before proceeding with accounts of further tests, I must here withdraw a suggestion made by me before this Society in November, 1880,‡ to the effect that starch glucose, mixed with refined

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\* "Journal of the American Chemical Society," Vol. II., p. 111.

† *Ibid*, p. 428.

‡ *Ibid*, p. 429.

cane sugar, could very likely be determined quantitatively by washing the suspected sugar with a saturated solution of starch glucose in methylic alcohol, and then weighing the residue. I suggested this process as one that might succeed, but one which I had not had occasion to try. The results which I have since obtained with this process show that the residue is much less than the quantity of starch sugar originally present. This is undoubtedly owing to this, that after dissolving cane sugar, the methylic solution acquires the property of dissolving additional quantities of starch sugar.

The sample of suspected sugar was also tested by the optical saccharometer. The direct test was 93.7 p. c. The solution was inverted, giving as a result 74.5 p. c. of pure sugar. This result clearly showed adulteration by a dextro-rotary substance. It does not, however, represent the actual quantity of cane sugar present, as I will show, by examining the influence of anhydrous glucose in a case of this kind.

To study this influence, I took 26.048 grammes of anhydrous glucose, which were dissolved in sufficient water to give 100 cc. of solution. A portion of this being placed immediately in the tube of the saccharometer, gave a deviation of 115.4 to the right. After standing in the tube for twenty minutes, the deviation fell to 95.7; after two hours, the deviation was 82.5. It was then allowed to stay over night in the tube, and, fourteen hours after, the deviation had fallen to 78.8, which did not vary afterwards on standing for several hours. The solution was then heated to 68° C. with 10 per cent. of its volume of hydro-chloric acid, the operation taking about fifteen minutes. This, after being neutralized with carbonate of soda gave a deviation to the right still equal to 78.8. There had then been no change by treating with hydro-chloric acid, which shows that the presence of dextrose is no bar to obtaining correct results by inversion, provided both observations, before and after inversion, are made after the deviating power of dextrose has reached a state of rest.

Returning to the test of the suspected sugar by the optical saccharometer, we have seen that the deviation to the right was at first 93.7 per cent. After standing in the tube fifteen minutes, the deviation fell to 90.2. We have seen that the result obtained by inverting, after the first reading, was 74.5. This result is evidently too high as the dextro rotary power of the dextrose present had diminished in the interval between the two observations.

Another sample of adulterated sugar was placed in the sacchar-



ometer as quickly as was practicable, after dissolving it in water. The indication was 100.4 per cent. As this sugar had all the appearance of a low sugar, this indication was sufficient to show that it was adulterated. This solution was kept in the tube for further examination. Fifteen minutes afterwards, the deviation had fallen to 94.3 per cent. Half an hour from the first observation, the reading was 91.6 ; one hour after, 90.2 ; two hours after, 89.9 ; three hours after, 89.7 ; five hours after, 89.3. After this there was no further variation. After inversion the indication was 72.7 per cent., which was considered as the correct percentage of pure sugar.

The optical saccharometer may then be considered as sufficient to determine if starch glucose is present in a sugar, by observing the deviation immediately after getting the solution ready for the saccharometer, and repeating the observation at sufficiently wide intervals of time. If dextrose is present in notable quantities, the deviation will decrease in a marked manner. Although the proof obtained in this way is satisfactory, it can only be applied by the very few persons who have saccharometers. To combat adulteration the best processes are those which can be easily applied by dealers and consumers, who are unprovided with scientific appliances.

The following process can be applied by using such means as are at the command of everybody. It is applicable to the detection of adulteration with either anhydrous or hydrated glucose :

Take two beaker glasses or two tea-cups. In one put a quantity of the suspected sugar, and in the other put about the same quantity of a sugar known to be refined sugar, free from adulteration.

Add, cautiously and gradually, a quantity of water to each sugar sufficient to make each equally and decidedly moist, and stir the sugar to mix it well and get it uniformly wet. Then place both cups in hot water. Any temperature between 50° and 100° C. will do. In about ten minutes the pure sugar will appear more moist than when cold, while the other sugar, if it contains a sufficient amount of starch glucose, will have sunk into a pasty, sticky mass, analogous to the *fill mass* of sugar refiners.

The application of heat is not indispensable, as a difference may be obtained by allowing the two sugars to stand several hours after being moistened, but, with heat, the effect is immediate, and is much more marked.

If the two samples of sugar are allowed to stand in the cups after they have cooled down, the pure sugar will look dryer on becoming

cold, while the adulterated sugar will continue in the state of a pasty, sticky mass.

This test is founded on the property possessed by cane sugar to form viscous, uncrystallizable compounds when mixed with many organic or inorganic substances, among which are anhydrous and hydrated dextrose. An example of compounds of this kind is *molasses*, obtained as a residuum in sugar manufacture.

As long as a mixture of cane sugar and starch glucose is sufficiently dry, it may look fair enough, as the elements which form molasses are kept from combining by want of water. The sugar adulterator is well aware of this, and he is careful to dry his sugar before mixing with glucose. Indeed, one characteristic of adulterated sugars is that they are always dryer than refined sugars of the same grade, which are known as *coffee sugars*, and are always sold moist.

As soon as sufficient water is added to an adulterated sugar, and moderate heat is applied, enough viscous syrup is formed to make the sugar sink into a paste, which remains permanently soft.

Useful indications may be obtained as to adulteration by starch glucose, by means of Fehling's solution. An ordinary refined coffee sugar will rarely show more than 5 percent. of glucose, while a sugar adulterated with the usual dose of starch glucose, will show about 20 per cent. of glucose.

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## ANALYSIS OF SOAP.

By DR. ALBERT R. LEEDS.

In the analysis of soap it is necessary to determine :—

- (1) Water.
- (2) Uncombined fat.
- (3) Soap consisting of (3a.) combined Fatty Acids, estimated as Fatty Anhydrides, and (3b.) Combined Alkali, usually Soda ( $\text{Na}_2\text{O}$ ).
- (4) Uncombined Alkali.
- (5) Glycerine.
- (6) Resin.
- (7) Sodid Carbonate.
- (8) Sodid Chloride.
- (9) Sodid Sulphate.

(10) **Sodic Silicate**—consisting of (10a) Soda combined in Silicate, and (10b.) Silica.

(11) **Starch.**

(12) **Insoluble Residue**, or mineral impurities, such as talc, clay, ochre, sand, etc.

(1) *Water*.—Weigh out about 5 grms. in very fine, small shavings upon a dried, weighed, plaited filter. Dry at  $110^{\circ}$  until weight is constant. The loss is water.

(2) *Uncombined fat*.—Transfer the filter containing the dried soap to the funnel connected with the return cooler as in the determination of the albuminoids in milk, and connect with the funnel a small tared flask containing 50cc. petroleum ether. After complete extraction distil off the ether, and the residue in the flask dried at  $110^{\circ}$  will be the uncombined fat.

(3) Soap, (4) Free Alkali, (5) Glycerine. Allowing the funnel with the soap freed from moisture and from fat to remain on the return cooler, attach to it a flask containing about 75cc of 95% alcohol and extract. To the alcoholic solution add a few drops of phenol-phthalëin, if free alkali be present neutralize with normal sulphuric acid and calculate the amount of uncombined soda.

After neutralization add a large excess of water and boil off the alcohol. To the aqueous solution, add a large excess of normal sulphuric acid. Boil, cool and decant through a small filter, wash with hot water and decant after cooling through the filter until litmus paper is no longer reddened by the washings. The filtrate consists of the combined soda and glycerine; the residue of fatty acids and resin. Neutralize the filtrate with normal soda solution and calculate the amount of combined soda as  $\text{Na}_2\text{O}$ . Evaporate to dryness and extract the glycerine with absolute alcohol. Transfer the alcoholic solution to a tared flask, distil off the alcohol, dry at  $100^{\circ}$  and weigh the residue as glycerine.

**Fatty acids and Resin.** Dissolve the small amount of the fatty acids and resin that may be on the filter, through which the decantation was effected, with a little petroleum ether, add the solution to the larger bulk in the beaker, evaporate off the ether, dry at  $110^{\circ}$  and weigh the combined fatty acids and multiply this result after subtracting the amount of resin by 0.97, and the product is the Fatty Anhydrides and resin.

(6) **Resin.** The resin was separated from the fatty acids according to the method proposed by Gladding (*American Chemical Journal*, Vol. III, p. 416.)

The original article by Gladding, reads as follows :—

### THE QUANTITATIVE SEPARATION OF ROSIN FROM FATS.

“About 0.5 gram of the fat acids containing the rosin is introduced into a small flask. Twenty cubic centimetres of 95 per cent. alcohol are added, and the flask rotated till the fat acids and the rosin are dissolved. A drop of phenol phthalëin is now added, and then a saturated solution of caustic potash in alcohol drop by drop, with thorough agitation after the addition of each drop, until the deep red color characteristic of alkalinity is obtained. One or two additional drops are now added, and the flask placed on a water-oven and kept at the temperature of boiling alcohol for ten minutes to ensure the saponification of the last portions of fat. The flask should be loosely corked. It is now cooled and the contents washed into a graduated 100 cc. cylinder by means of concentrated ether. The cylinder is filled with the ether exactly to the 100 cc. mark ; then corked, best by a common cork twisted tightly in, and the contents mixed by a moment's shaking. About one gram of C. P. *neutral* silver nitrate is now rubbed to an impalpable powder in a small mortar and then introduced into the cylinder. The latter is vigorously shaken for ten or fifteen minutes until the flocculent precipitate of silver stearate and oleate collects in the same manner as silver chloride upon shaking, and settles clear. Fifty to seventy cc. of the supernatant liquid are now syphoned off by means of a slender syphon, previously filled with ether, into a second 100 cc. cylinder, passing the liquid through a small filter paper if it is not perfectly clear. A small quantity of pulverized silver nitrate is shaken up with this to make certain that all the oleate and stearate are precipitated. If no flocculent precipitate appears this is the case. Twenty cc. of a mixture of hydrochloric acid and water, one-third the former and two-thirds the latter, are now added, and the cylinder vigorously shaken until the decomposition of all the silver salt present is complete. After the silver chloride has settled, an aliquot portion of the supernatant ether solution is syphoned off into a platinum dish and evaporated on the top of a water-oven to dryness. The residue is rosin containing a small amount of oleic acid, which can be accurately allowed for.”

The following experiment gives the allowance to be made :

“*Exp. II.* One-half a gram of pure fat acid was treated in the above way. Eighty cc. shaken with HCl and evaporated gave

0.0188 gram of residue ; 10 cc. contain therefore 0.00235 gram of oleic acid. This very small and accurate correction of 0.00235 gram for every 10 cc. of liquid shaken up with HCl was applied in the subsequent experiments."

The manner of calculating the allowance may be most clearly shown by another experiment :

"*Exp.* IV. One-half a gram of the fat acids from linseed oil was add to 0.050 gram of rosin and treated as the above.

"Seventy cc. shaken with HCl gave 0.0517 gram of residue. Subtracting the allowance for 70 cc., namely, 0.0164 gram, leaves 0.0353 gram of 0.0504 gram of rosin on the whole quantity taken instead of 0.050 gram taken."

About 0.5 gram. of the mixture of the fatty acids and resin are dissolved in twenty cc. of strong alcohol and, with phenol-phthalëin as an indicator, soda is run in to a slight supersaturation. The alcoholic solution, after boiling for ten minutes to ensure complete saponification, is mixed with ether in a graduated cylinder till the volume is 100 cc. To the alcoholic and ethereal solution 1 gram. of very finely powdered Ag. NO<sub>3</sub> is added and the contents of the cylinder are shaken thoroughly for ten or fifteen minutes. After the precipitate has settled, 50 cc. are measured off and if necessary filtered into a second graduated cylinder. A little more Ag. NO<sub>3</sub> is added to see if the precipitation is complete and then 20 cc. of dilute hydrochloric (1:2) to decompose the silver resinate. An aliquot part of the ethereal solution in the cylinder is evaporated in a tared dish and weighed as resin, deducting a small correction (for 10 cc. 0.00235 gram.) for oleic acid. The amount of resin subtracted from the combined weight of fatty acids and resins, as found before, gives the fatty acids. (7) Sodid carbonate ; (8) Sodid chloride ; (9) Sodid sulphate ; (10) Sodid silicate ; (11) starch ; (12) insoluble residue. The filter in the funnel connected with the return cooler, after treatment with alcohol contains the mineral constituents of the soap. The contents of the filter are washed with cold water till the washings amount to 60 cc. The filter is then dried and weighed. The weight gives the insoluble residue and starch.

The starch is converted into C<sub>6</sub>H<sub>12</sub>O<sub>6</sub> with dilute acid and titrated with Fehling's solution. The weight of starch found subtracted from the total weight of insoluble residue and starch gives the insoluble mineral constituents.

The aqueous solution of 60 cc. just mentioned, is divided into

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The starch is converted into C<sub>6</sub>H<sub>12</sub>O<sub>6</sub> with dilute acid and titrated with Fehling's solution. The weight of starch found subtracted from the total weight of insoluble residue and starch gives the insoluble mineral constituents.

The aqueous solution of 60 cc. just mentioned, is divided into

four equal parts, in one of which is determined the carbonate of soda titration and in the other parts, the chloride, the sulphate and the silicate respectively, by any convenient method.

My thanks are due to my assistant, Dr. Edgar Everhart, for his assistance in working out the details of this scheme.



**W**elch out 5 grms. Dry at 110°. Loss corresponds to water. Treat with petroleum ether.

<p><b>Residue</b> = Soap and Mineral constituents. Treat with alcohol.</p>	<p><b>Residue.</b> -Na<sub>2</sub>CO<sub>3</sub>, NaCl, Na<sub>2</sub>SO<sub>4</sub>, sodium silicate, starch and insoluble residue. Wash with 60 cc. water.</p>	<p><b>Residue</b>—Starch and Insoluble</p>	<p><b>Filtrate</b> Na<sub>2</sub>CO<sub>3</sub>, NaCl, Na<sub>2</sub>SO<sub>4</sub>, and Sodium silicate. Divide into 4 equal parts.</p>	<p><b>Na<sub>2</sub>CO<sub>3</sub>, Titrate with normal H<sub>2</sub>SO<sub>4</sub>, and calculate as Na<sub>2</sub>CO<sub>3</sub>.</b></p>	<p><b>NaCl Titrate with AgNO<sub>3</sub>, or weigh as AgCl. Calculate as NaCl.</b></p>	<p><b>Na<sub>2</sub>SO<sub>4</sub>, Weigh as BaSO<sub>4</sub>, Calculate to Na<sub>2</sub>SO<sub>4</sub>.</b></p>	<p><b>Sodium silicate. Decompose with HCl, and determine soda combined in Silicate and Silica.</b></p>	<p><b>Insoluble Residue.</b> Dry the starch filter. The weight is the starch and</p>	<p><b>Convert the starch into C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>, Titrate with Fehling's solution. Subtract weight of starch found and difference is Insoluble Mineral constituents.</b></p>
<p><b>Extract</b> = Soap (Fatty anhydride, resin and combined alkali), Glycerine and Free alkali. Add two or three drops of phenolphthalein. If necessary, titrate with normal H<sub>2</sub>SO<sub>4</sub>.</p>	<p><b>Residue</b> = Soap and Mineral constituents. Treat with alcohol.</p>	<p><b>Residue</b>—Fatty acids and Resin. Dry at 110° and weigh. Dissolve an aliquot part in 20 cc. strong alcohol, and using phenolphthalein as an indicator, saponify with soda in slight excess. Boil, cool and add either to 100 cc. Decompose with AgNO<sub>3</sub>, by adding in fine powder, and shake well for 10 minutes. Allow to settle.</p>	<p><b>Residue</b>—Fatty acids and Resin. Dry at 110° and weigh. Dissolve an aliquot part in 20 cc. strong alcohol, and using phenolphthalein as an indicator, saponify with soda in slight excess. Boil, cool and add either to 100 cc. Decompose with AgNO<sub>3</sub>, by adding in fine powder, and shake well for 10 minutes. Allow to settle.</p>	<p><b>Na<sub>2</sub>CO<sub>3</sub>, Titrate with normal H<sub>2</sub>SO<sub>4</sub>, and calculate as Na<sub>2</sub>CO<sub>3</sub>.</b></p>	<p><b>NaCl Titrate with AgNO<sub>3</sub>, or weigh as AgCl. Calculate as NaCl.</b></p>	<p><b>Na<sub>2</sub>SO<sub>4</sub>, Weigh as BaSO<sub>4</sub>, Calculate to Na<sub>2</sub>SO<sub>4</sub>.</b></p>	<p><b>Sodium silicate. Decompose with HCl, and determine soda combined in Silicate and Silica.</b></p>	<p><b>Insoluble Residue.</b> Dry the starch filter. The weight is the starch and</p>	<p><b>Convert the starch into C<sub>12</sub>H<sub>22</sub>O<sub>11</sub>, Titrate with Fehling's solution. Subtract weight of starch found and difference is Insoluble Mineral constituents.</b></p>
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## ABSTRACTS.

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Abstracts from *Berichte der Deutschen Chemischen Gesellschaft*, by Percy Neymann, Ph. B.

**On Synthetical Oxalic Acid.** V. MERZ and W. WEITH, (Vol. XV., p. 1507). The idea was taken from the fact that formic acid can be produced from inorganic material, namely, carbonic oxide and caustic soda. Considering the large production of alkali formiates, it seemed possible that oxalic acid might be formed from these materials. Investigation was extended to the potassium and sodium salts of formic acid, also the calcium, barium and magnesium salts. The formiates were subjected to the heat of baths of various substances. In the diphenylamin bath ( $310^{\circ}$ ) the sodium formiate is scarcely decomposed. At  $360^{\circ}$  (mercury bath) it is mostly converted into carbonate, and at  $440^{\circ}$  (sulphur bath) it is mainly converted into the oxalate. The potassium salt behaves in a similar manner. The other salts gave no oxalate even at  $440^{\circ}$ .

**Vanadintrichlorid from Vanadintrisulphide.** W. HALLERSTADT, (Vol. XV., p. 1619.) The sulphide is heated (in a drawn out piece of combustion tubing, the small end of which is connected with a distilling flask) in a current of chlorine at a moderate temperature. The liquid produced is distilled. The residue, which consists of Vanadintrichlorid, is treated in a steam of carbonic acid at  $150^{\circ}$  C.

**An Ammoniacal-alkaline Silver Solution as Reagent for Aldehyde.** B. TOLLENS, (Vol. XV., p. 1635.) The author finds a solution of the following composition to be the best :

Three grms. of nitrate of silver are dissolved in 30 grms. of ammonia of 0.923 sp. gr.

Three grms. of caustic soda are dissolved in 30 grms. of water. The two solutions are mixed and used after a few days. The reagent must be kept in a cool, dark place.

**Quantitative Estimation of Fusil Oil in Brandy.** L. MARQUARDT, (Vol. XV. p. 1661.) Continued from Vol. XV. p. 1370. Author adds that ether cannot be used in place of chloroform. Common chloral chloroform, such as is used for surgical purposes is not pure enough. Even that known as pure chloroform should be subjected to one or two oxidations with bichromate of

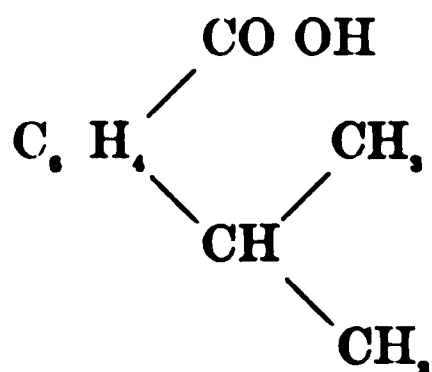
potassa and sulphuric acid. The alcohol must also be purified. The author appends a qualitative analysis.

**On Soluble Alizarin Blue.** H. BRUNCK and C. GRAEHL, (Vol. XV. p. 1783.) The dye is called alizarin blue S. alizarin blue as it occurs in commerce, in a paste of 10—12 % of dry constituents is mixed with a 25—30 % solution of sodium bisulphite. When the solution is filtered, unaltered alizarin blue remains on the filter which can be treated again. The filtrate contains the new compound which can be obtained in crystalline form.

**Preparation of Peroxide of Lead.** A. FEHRMANN, (Vol. XV. p. 1882.) It is best and most cheaply prepared by treating a solution of chloride of lead (50—60° C.) with chloride of lime until a filtered sample shows no more brown color. It is then filtered off with exclusion of air.

**The Synthesis of Cuminic Acid.** RICH. MEYER and ERWIN MUELLER, (Vol. XV. p. 1903.) The cumol was prepared according to Gustavson's method, by the action of secondary propylbromid on benzol in the presence of  $Al_2Br_3$ .

The synthetical cumol was transformed into the bromine compound in the usual manner, and the acid produced in the same manner as in the first experiments (Vol. XV. p. 496.698, Ber-d-d-chem Ges.) The melting point was found to be 116°—117° C., and the formula (1.4) :



**On the Reduction of Molybdenum Compounds.** OTTO FREIHERR VON DER PFORDTEN, (Vol. XV. p. 1925.) The volumetric method is important. Titration is performed in the usual manner, whereby the suboxide is transformed into the sesquioxide which latter is used for calculations. The salt is dissolved in a little water, to which are added 60 cc of a 27 per cent. hydrochloric acid and 8—10 grms. of zinc in bars, the iron in which has been determined by titration. When the solution has assumed a yellow color, and before all the zinc is consumed the flask is cooled, and contents thrown into a porcelain dish containing water, dil. sulphuric acid and manganous sulphate solution of the concentration

given by *Zimmermann*. From a pipette more permanganate solution is added and titred to pink color as usual. The results are accurate

$$1 \text{ cc K Mn O}_4 = 0.000752185 \text{ Oxygen}$$

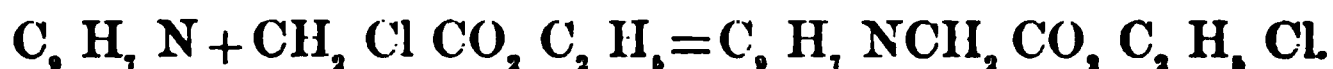
$$1 \text{ cc K Mn O}_4 = 0.00451311 \text{ Mo O}_3$$

**Method for determination of phosphoric acid.** *Otto, Freiherr v. d. Pfordten.* (Vol. XV., p. 1929).

The method is based upon the estimation of molybdic acid. The composition of the yellow precipitate is assumed to be that given by Finkener and Pemberton. One mol.  $\text{P}_2\text{O}_5$  to 24 mol.  $\text{MoO}_3$ .

The method is applicable in all cases in which the separation of the phosphoric acid with molybdate of ammonium can take place.

**On some chinolin derivatives.** *O. Rhoussopoulos.* (Vol. XV., p. 2008). Chinolin and Ethylmonochlor-acetate give rise to a chloride.



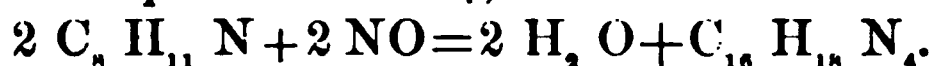
It may be termed the chlorohydrate of the chinolin-glycocollethylether.

The Betain of Chinolin  $\text{C}_{11}\text{H}_9\text{NO}_2, \text{H}_2\text{O}$  can be obtained from the chlorohydrate by treatment with oxide of silver.

**On the application of diphenylamin and anilin in qualitative analysis.** *Conrad Laar.* (Vol. XV., p. 2086).

**On azylines, a homologous series of bases containing nitrogen.** *E. Lippmann and F. Tleisner.* (Vol. XV., p. 2136).

Nitrous oxide acts on amines and amido acids, eliminating nitrogen and forming a hydroxyl derivative. When nitrous oxide is passed into an alcoholic solution of dimethyl anilin, the solution is colored deep red, carbonic acid is evolved and crystals are formed. The reaction takes place according to the formula :



Azylines are compounds containing nitrogen which are obtained by the action of nitrous oxide on bases into which the  $=\text{N}-\text{N}=\text{N}$  has entered the benzol ring with four free bonds while azo compounds are those which are characterised by the bivalent group  $-\text{N}=\text{N}-$ .

The compounds are insoluble in water but soluble with beautiful red color in hydrochloric acid and with a beautiful green color in acetic acid. The melting points increase with the decrease in molecular weight.

**Contributions to the history of conydrin.** A. W. HOFMANN. (Vol. XV., p. 2313). The author determines the formula of conydrin to be the same as that given by *Wertheim*  $C_8H_{17}NO$ .

**On the behavior of the alkali phosphates towards some indicators.** G. TOBIAS. (Vol. XV., p. 2452). By the action of phosphoric acid on alkalies the changes of color of the indicators are not as decided as with other acids. Experiments with litmus, phenolphthalëin and aqueous solution of cochineal on different solutions (1) a dilute aqueous solution of pure commercial phosphoric acid was taken and allowed to stand for some time, it then was boiled to remove all modifications of phosphoric acid, except ortho—100 cc contained 4.095  $H_3PO_4$ ; (2) a solution of double this strength, 100 cc = 8.190  $H_3PO_4$ ; (3) potassa, freed from carbonic acid and from  $K_2SO_4$  by means of  $Ba(OH)_2$ , 100 cc = 4.096 KOH; (4) soda solution, 100 cc = 2.349 NaOH; (5) ammonia, freed from carbonic acid, 100 cc = 1.950  $NH_3$ ; (6) di-sodic phosphate, 100 cc = 3.4165  $Na_2HPO_4$ .

In no case were the results satisfactory, the indicators mentioned above should not be used for the titration of phosphoric acid.

**Lecture experiments with Zinc dust and Sulphur.** II. Schwarz. (Vol. XV., p. 2505). The old experiment employed for demonstrating chemical union by heating sulphur and copper or iron filings is well known. The demonstration can be shown much more brilliantly by heating a mixture of zinc dust and sulphur. When mixed in the proportion of atomic weights, 65 parts of zinc and 32 parts of sulphur in a mortar (but with great care) the mixture can be easily ignited by a match and will burn like gunpowder. The mixture can be partly exploded by a smart blow of the hammer.

When the vapor of bisulphide of carbon is brought in contact with heated zinc dust, the zinc dust immediately begins to glow; sulphide of zinc is formed and carbon separates.

Carbon in statu nascendi easily unites with nascent hydrogen. A mixture of sulphuretted hydrogen and bisulphide of carbon vapor is conducted over zinc dust, which is gently heated. The resulting gas after passing through KOH to absorb undecomposed sulphuretted hydrogen, was found to consist of hydrogen and large quantities of marsh gas.

**On the Demonstration of the Flashing Point of Petroleum.** J. T. STODDARD. (Vol. XV., p. 2555). Description of apparatus, which is very simple and can be made easily in any laboratory.

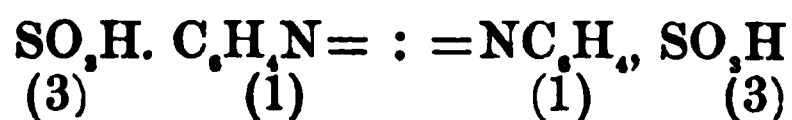
**Contributions to the Knowledge of the Direct Substitution Products of Azobenzol.** H. JANOOSKY. (Vol. XV., p. 2575). The majority of the substitution products of azo-benzol which have been produced within late years were obtained indirectly by reduction of substituted nitro-compounds or by oxidation of substituted amido-compounds, or by diazotation and subsequent combination. By sulphurizing with fuming sulphuric acid of 10-14 % azobenzol-mono-sulpho-acid is produced ( $C_{12}H_9N_2SO_3H + 3 \text{ aq.}$ ). By decomposing with iron and acetic acid anilin and amido-benzol-para-sulpho-acid are produced. The formula of the mono-sulpho-acid would hence be  $C_6H_5N= : =NC_6H_4SO_3H$ .

$$\begin{matrix} (1) & & (4) \end{matrix}$$

Three acids are obtained by treating one part of azobenzol with four parts of cryst. acid of 20-30 %. The first or alpha-acid is  $C_{12}H_9N_2(SO_3H)_2 + 3 \text{ aq.}$ , or  $SO_3H.C_6H_4N= : =NC_6H_4SO_3H$ . It is identical with the acid produced from the nitro-sulpho-acid of benzol (1.4) or from the amido-sulpho-acid (1.4).

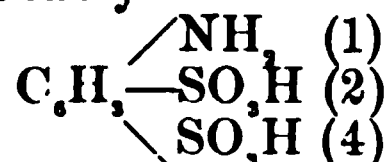
$$\begin{matrix} (4) & & (1) & & (1) & & (4) \end{matrix}$$

The second acid is an amido-benzol-meta-sulpho-acid of the formula :



It crystallises with five molecules of water of crystallisation.

The third acid is probably



By decomposition it yields anilin and amido-benzol-disulpho-acid.

**On the Recognition and Estimation of Titanium.** A. WELLER. (Vol. XV., p. 2592). The author recommends peroxide of hydrogen as a reagent in testing for titanous acid. A few drops of the peroxide added to 1cc of a sulphuric acid solution of titanous acid will produce an orange-red color, when as much as one milligramme of the acid is present, and a distinct light yellow color when 0,0001 grms. is present. Only when the amount is equivalent to 0,0002 grms. the reaction ceases to be distinct. This reaction is employed for a quantitative colorimetric determination of titanium and gives very good results in as much as the titanium can be determined in the presence of zirconium, tantalum and niobium. When, however, vanadic or molybdic acid is present, great care must be exercised, as these acids are also colored when  $H_2O_2$  is added to a solution containing them.

# ACTION OF DIAZOANISOLE UPON PHENOLS.

BY JAMES H. STEBBINS, JR., F. C. S.

## SECOND PAPER.

In my last paper on this subject, I described a compound produced by the action of diazoanisoie chloride, upon an alkaline solution of orcine.

At present it is my intention to describe a somewhat similar body resulting from the union of diazoanisoie chloride, and resorcine disulphonate of soda.

12. 3 g. anisidine were diazotized with 17.9 g. H Cl (1.20 Sp. Gr.) and 6.9 gr. Na NO<sub>2</sub>, in 200 c. c. water.

The diazoanisoie chloride thus formed, was added, little by little, and under constant stirring to a solution composed of 18.9 grs. resorcine disulpho acid, 16 grs. Na OH in 200 c. c. water, and 20 c. c. Na OH and Aq. (10 per cent.)

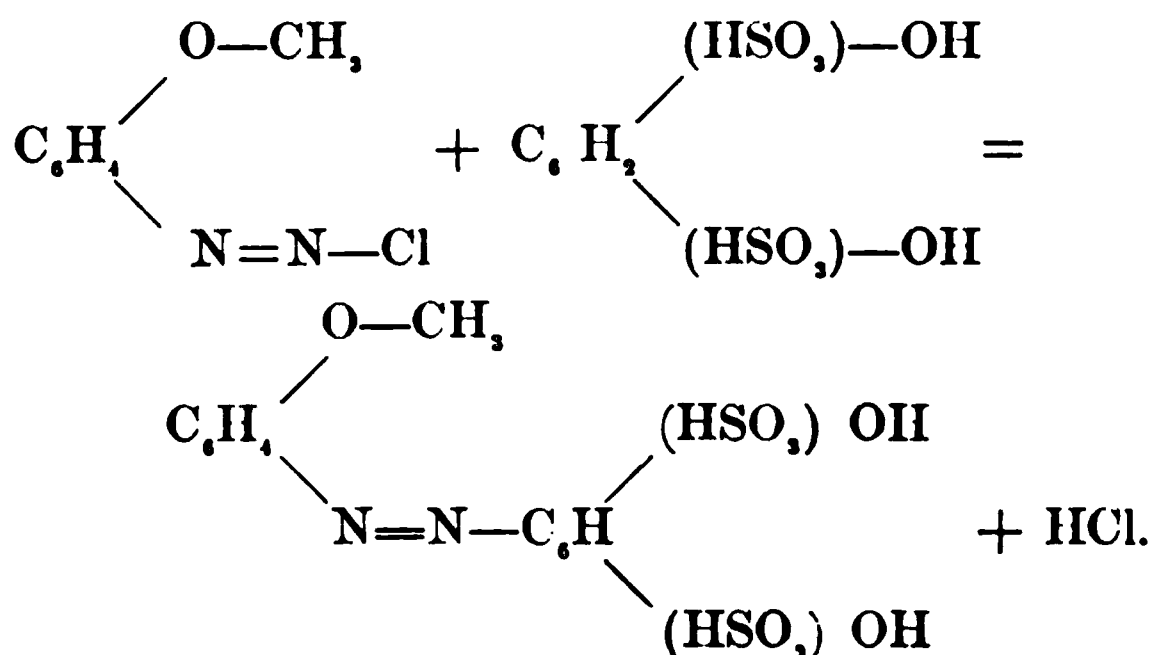
The whole was allowed to rest for a few hours, and then treated with an excess of strong muriatic acid, which precipitated the dye-stuff as a free acid.

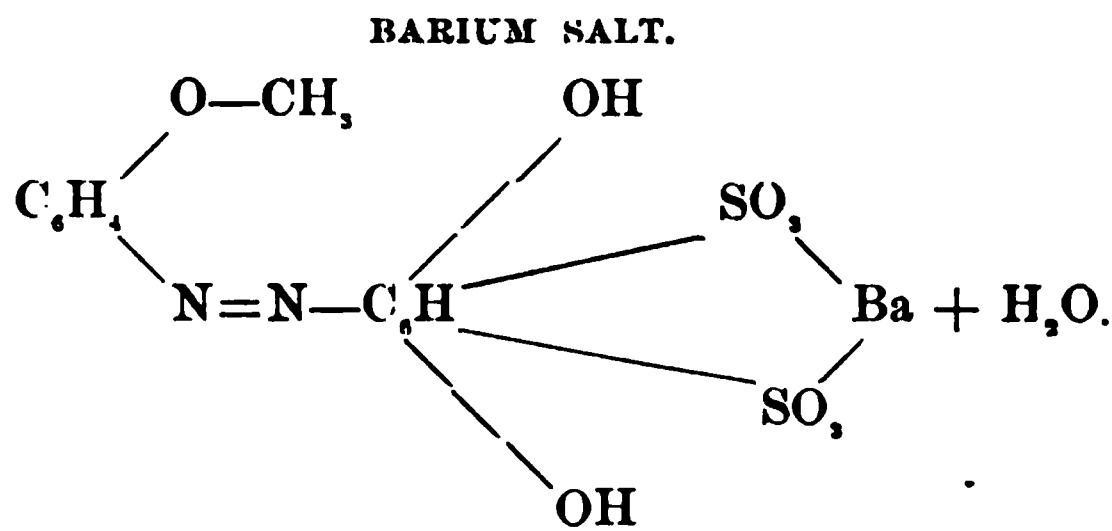
It was then dissolved in hot water and allowed to crystallize.

Thus prepared it crystallizes in little fern shaped yellow plates, which are easily soluble in hot and cold water.

### RESORCINE-DISULPHO ACID AZO-ANISOLE.

This substance is probably formed according to the following equation :





This interesting salt, was obtained, by dissolving the free acid in hot water and treating with an excess of Ba Cl<sub>2</sub>.

The precipitate formed was collected on a filter, washed and dissolved in boiling water, from which it crystallized on cooling in fine, yellow, star-grouped needles.

In transmitted light this salt has a yellow color, but in reflected light a brick-red shade.

It is sparingly soluble in cold, but readily soluble in boiling water, from which it crystallizes with one molecule water of crystallization.

A barium estimation gave :

Barium.....24.56%

THEORY.

Barium.....24.56%

ON A NEW DEVICE FOR DETERMINATION OF CARBON IN CAST IRON.

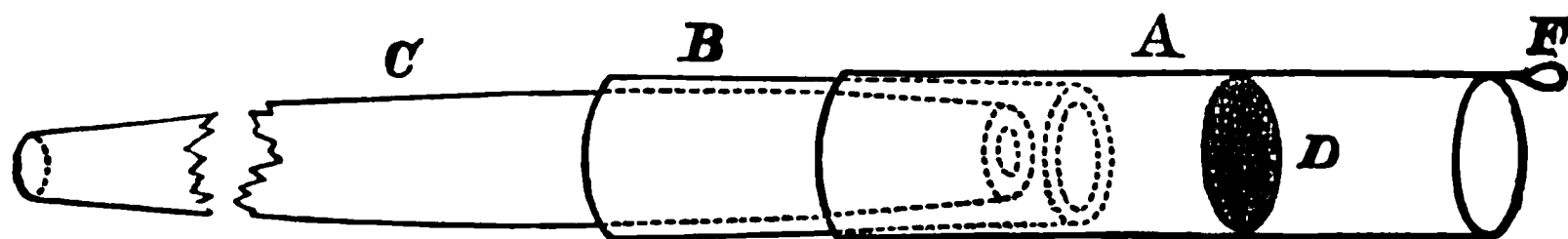
BY A. A. BRENNEMAN, S. B.

The carbonaceous residue obtained when cast iron is dissolved is commonly transferred with the mass of asbestos serving as a filter to the combustion tube. To avoid accidents incident to this transfer, and to ensure the combustion of refractory portions of graphite, the writer has adopted the modifications of the process described below. A brief note upon the process was read at the meeting of the American Association in 1879, but the success attending its use since then by students under the writer's direction, as well as the introduction of some minor improvements, has led him to believe that a fuller description of the process might be useful to others. The process depends in the main upon the use of combustion tubes of porcelain, and the employment of a small tube of platinum as a



filtering tube, which is subsequently put with its contents into the combustion tube and heated in a stream of oxygen in presence of cupric oxide in the usual way.

The details of the filtering apparatus will be best understood from the figure.



A is a tube of moderately thick platinum foil, somewhat thicker than that used in blowpipe work. B is a piece of soft rubber tubing fitting snugly within A. C is a piece of glass combustion tubing, tapered at each end as shown, and having a length of about 12 c. m. Other dimensions are as shown in the figure, viz., A  $45 \times 13$  m. m., B 40 m. m. in length. A may vary in dimension according to circumstances.

The apparatus is connected together by pulling or pushing the tube B over the tapering end of C after it has been loosely inserted in A. All pressure upon A is thus avoided. The joint is air-tight. When not in use A is slipped over a piece of glass tubing of proper size to protect it from injury. The loop F serves to introduce a slightly hooked glass rod by which A is moved into the combustion tube or withdrawn from it. The disc D is of platinum foil, perforated, and when in use rests on the end of B within A, and supports the asbestos used in filtration. A small funnel C serves to convey liquids to the filtering tube. The filter with its contents when dry is separated from B and C and introduced into the combustion tube, where it is heated in a current of oxygen as mentioned, in the usual way.

The tube A is easily made in the laboratory, being shaped over a piece of combustion tubing and soldered with gold leaf. It should fit snugly within the porcelain tube, so that oxygen may pass mainly *through* the filter and its contents. It is not difficult to find porcelain tube of a proper size to suit the platinum tube, and in case of difficulty the latter can be enlarged by rubbing it with a stout glass rod while held on a piece of combustion tubing or reduced in size by cutting and resoldering.

The use of porcelain tubes is of advantage not only because of the high temperature attainable, but also because the average *life* of such a tube is greater than that of a quantity of glass combus-

tion tubes costing an equal sum ; with the certainty of perfect combustion that is assured in their use, the convenience of seeing the substance under combustion disappears.

The tubes of platinum seem to suffer little in use. One of them has sufficed for about fifty combustions and is still perfectly good.

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## ABSTRACTS.

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Abstracts from the *Journal of the Chemical Society*, London, by Arthur H. Elliott, Ph. B., F. C. S.

**On Oxypropyltoluidine.** By H. FOSTER MORLEY, M. A., Fellow of University College. (Vol. XL., p. 387).

Propylene oxide was dissolved in an equivalent of paratoluidine, and heated for hours on a water bath. On distilling no propylene oxide was obtained, but at  $285^{\circ}$ – $288^{\circ}$  a liquid was obtained, which afterward solidified and by crystallization from benzene gave the formula  $N(C_3H_7O)(C_7H_7)H$ . A better yield is obtained when the toluidine solution of propylene oxide is allowed to stand some days at ordinary temperatures. In the latter case 20 grms. of the base were obtained from 46 grms. of toluidine. Oxypropyltoluidine melts at  $74^{\circ}$  and boils at  $293^{\circ}$ . It is insoluble in water, soluble in benzene, ether, alcohol, and petroleum. Dissolved in solution of oxalic acid it gives crystals of the formula  $C_{10}H_{15}NO, H_2C_2O_4$ , which melt at  $151^{\circ}$ . On heating the oxalate to  $150^{\circ}$  it melts and gives off water, carbonic oxide, and carbonic acid, leaving a syrup.

Author also gives a description of the distillation of oxypropyltrimethylammonium hydrate. This base (see *J. C. S.*, Vol. XXXVIII., p. 877) resembles neurine when heated giving trimethylamine, propylene-glycol with other liquids, and carbonic acid.

**On some Halogen Compounds of Acetylene.** By R. T. PLIMPTON, Ph. D. (Vol. XL., p. 391).

The acetylene was obtained by Jungfleisch's method from coal gas. By passing the acetylene through bromine the author obtained the tetrabromide and a solid  $C_2HBr_3$  melting at  $174^{\circ}$ . By treating the tetrabromide in alcohol with zinc powder the dibromide was made ; it boiled at 110–111 and at  $17^{\circ}$  was still liquid. Its specific gravity at  $0^{\circ}C$ . was 2.268. The di-iodide was made by passing acetylene over iodine wet with alcohol. Crystallized from alcohol, it

gives needles melting at  $73^{\circ}$ , and distilling without decomposition ; it boils at  $192^{\circ}$ .

Acetylene chloriodide was prepared by passing the gas over iodine monochloride. The crude product was washed, dried and distilled. The chloriodide obtained was a heavy liquid boiling at  $119^{\circ}$  and has the formula  $C_2H_2ClI$ . Acetylene chlorobromide is prepared by adding bromine to acetylene chloriodide under water, dissolving the separated iodine in sodium thiosulphate, drying, and distilling the liquid. It boils at  $81^{\circ}$ – $82^{\circ}$ , and has the formula  $C_2H_2ClBr$ . Warmed with alcoholic soda it gives a spontaneously explosive gas.

Acetylene bromide was made by shaking acetylene with bromine iodide in bottles from which the air had been previously abstracted. The crude black oil is washed with sodium thiosulphate and purified by distillation with steam. It is a heavy colorless liquid, boiling at  $150^{\circ}$  and solid at  $8^{\circ}$ .

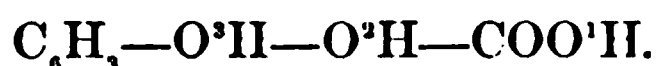
This paper is accompanied with a table showing the relation of the above compounds to those of ethylene and ethylidene.

**On Dihydroxybenzoic Acids and Iodo-Salicylic Acids.**  
By A. K. MILLER, Ph. D. (Vol. XL., p. 398).

In this research the author has obtained the sixth acid of the formula  $C_6H_2(OH)_2COOH$ . Two methods were employed ; one by heating catechol with ammonium carbonate and water in sealed tubes, and the other by fusing iodosalicylic acid with potassic hydrate. By the first method very little of the dihydroxybenzoic acid was obtained ; but it gave a *pure blue* coloration with ferric chloride, which turned violet red with sodium carbonate. Analysis gave figures for the formula  $C_6H_2O_4$ . For the second method the author made iodosalicylic acid by the direct action of iodine on salicylic acid. Lautemann, Lischti, Demole and Goldberg, worked upon iodosalicylic acids, but their results were so divergent that the author repeated the work. Having found that Lautemann's and Goldberg's work agreed in some points, the author made iodosalicylic acid by Lautemann's method, by boiling salicylic acid and iodine in alcohol. To separate mono- and di-iodosalicylic acids, the mixture was boiled with barium carbonate and water, and the barium salts crystallized from hot water ; but the method did not give definite salts and was abandoned. To effect the separation of two iodosalicylic acids a tedious process of fractional crystallization was undertaken. By this means the two acids were separated, the more insoluble melting at  $197^{\circ}$  and the other at  $198^{\circ}$ . The

first of these is paraiodosalicylic acid, it crystallizes from water in long needles, which give a violet color with ferric chloride. The barium salt of this acid crystallizes with 4 mol's of water and dissolves in 120–125 parts of water at 8°. The second more soluble acid obtained above is ortho-iodosalicylic acid; it gives a violet coloration with ferric chloride. The barium salt of the last acid requires 190 parts of water for its solution at 8°, and it crystallizes with  $3\frac{1}{2}$  molecules of water.

By fusing the para-acid with potash dihydroxybenzoic acid is obtained, which crystallized in anhydrous needles. Its aqueous solution gives *no* precipitate with lead acetate, gives a deep blue color with ferric chloride, changing to red with sodium carbonate, but destroyed by excess of the reagent. By fusing the ortho acid with potash, another dihydroxybenzoic acid is obtained, which gives a blue color with ferric chloride, but the color is violet red, with sodium carbonate, and not destroyed by an excess of the reagent. This acid also differs from the former in its crystalline form, by giving a precipitate with lead acetate, and by giving catechol and carbonic acid when heated, while the para-acid gives quinol under the same conditions. It melts at 204°, and is the missing dihydroxybenzoic acid.



The author gives a table of the six dihydroxybenzoic acids, with their properties, etc.

**Crystalline Molecular Compounds of Naphthalene and Benzene with Antimony Trichloride.** By WATSON SMITH and G. W. DAVIS. (Vol. XL., p. 411.)

By melting together three parts of antimony trichloride with two of naphthalene, minute clinorhombic tables are formed, which are separated from the fluid mass with a warm platinum spatula. When first obtained they are transparent, but soon become opaque. They gave by analysis 38.68 and 38.56 per cent. of antimony, corresponding to the formula  $3\text{Sb Cl}_3, 2\text{C}_{10}\text{H}_8$ , which requires 38.94 per cent. of antimony.

By dissolving three parts of crystallized antimony trichloride in four parts of benzene with warming; in a few days large inorhombic plates are formed, which are colorless and transparent, and remain so. Analysis gave 43.46 per cent. of antimony and 37.62 per cent. of chlorine, corresponding to the formula  $3\text{Sb Cl}_3, 2\text{C}_6\text{H}_6$ ,

which requires 43.57 per cent. of antimony and 37.90 per cent. of chlorine.

**An Additional Evidence, by Analysis of the Quinoline Molecule, that this Base Belongs to the Aromatic Series of Organic Substances.** By WATSON SMITH and G. W. DAVIS. (Vol. XL., p. 412.)

By heating quinolene with ten times its weight of antimony penta-chloride in a sealed tube, the author obtained the products of exhaustive perchlorination. Among these products they obtained perchlorethane as white needles melting at  $210^{\circ}$ , but probably containing perchlorbenzene. By resubliming, a product was obtained melting at  $182^{\circ}$ , pure perchlorethane melting at  $183^{\circ}$ . They also obtained perchlorbenzene as white needles, melting at the correct temperature,  $223^{\circ}$ . No perchlormethane was detected, but this might escape in opening the tubes. These results coincide with the work of Dewar (J. C. S., 1881, p. 1044), who obtained quinolinic acid by oxidizing quinoline, and by heating the acid with soda-lime got aniline, the benzene nucleus with an amido-group; while the present authors obtain the benzene nucleus perchlorinated, and half the remaining residue, containing the nitrogen atom, as perchlorethane.

**On Orcinol and Some of the Other Dihydroxytoluenes.** By R. H. C. NEVILE and DR. A. WINTHER. (Vol XL., p. 415.)

Believing that Orcinol was a dihydroxytoluene, the authors undertook this investigation. Taking dinitroparatoluene 1:3:5 made from dinitropartoluidine, it was dissolved in alcohol and treated with ammonium sulphide. Water was added and the precipitate dissolved in hydrochloric acid and reprecipitated with ammonia. By further purification thick needles of nitrotoluidine were obtained melting at  $98^{\circ}$ – $98^{\circ}.4$ . The yield was 50 per cent. of the dinitrotoluene used. By treating with sulphuric acid the sulphate was obtained; which by further treatment with potassic nitrite gave nitrocresol as a brown oil, which by purification with ether gave yellow needles of nitrocresol. The nitrocresol was treated with tin and hydrochloric acid; after removing the tin, the solution was evaporated, and the crystals obtained washed with ether to remove unreduced nitrocresol. From this chloride dihydroxytoluene is obtained by treating with sulphuric acid and potassic nitrite. The resulting oil purified by distillation and crystallizing from benzene and chloroform, gives

from the first needles or prisms and from the latter white leaves. After 14 days over sulphuric acid it melts at  $106^{\circ}$ – $108^{\circ}$ ; which by analysis proves to be orcinol  $C_6H_2CH_3(OH)_2$  1:3:5.

The authors have also succeeded in making orcinol from meta-bromo-toluene–metasulphonic acid; toluene–meta-disulphonic acid; metabromo–metatoluidine; metadibromotoluene.

During this research the authors have also obtained the dihydroxytoluene 1:2:4, starting with orthonitro-paratoluidine. This latter was converted in sulphate, treated with potassic nitrite and converted into nitrocresol. This nitrocresol was treated with tin and hydrochloric acid to obtain amido-cresol. The chloride of amido-cresol thus obtained by treatment with sulphuric acid and potassic nitrite and the dihydroxytoluene obtained as an oil, which afterward crystallized, and by sublimation and crystallization gave a melting point of  $104^{\circ}$ – $105^{\circ}$ .

The author also prepared dihydroxytoluene 1:2:5 from meta-nitro-orthotoluidine following nearly the same process as in above case. The dihydroxytoluene 1:2:5 obtained is in white leaves melting at  $124^{\circ}$ – $125^{\circ}$  and freely soluble in alcohol, ether or water.

An investigation of the dihydroxytoluene 1:3:4 was also made by the authors starting with metanitro-paratoluidine, following the method above used by preparing the diazo compound, but was unsatisfactory. The method of Wagner (Ber. 1874, 537) was used, and by slight modification with success.

The paper is a very lengthy one and filled with most interesting details in manipulation of the methods used.

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Abstracts from *Berichte der Deutschen Chemischen Gesellschaft*, by Percy Neymann, Ph. B.

**On Hydroxylamin Chloride.** V. MEYER. (Vol., XV. p. 2,789.) The author gives a method which can be used on a large scale without the use of platinic chloride. He finds that the presence of ammonium chloride does not impair the durability of the product, but the presence of ferric chloride or free acid cause decomposition of the product, forming ammonium chloride.

**On the Behavior of Platinum or Palladium Towards Carbonic Oxide or Hydrogen in the Presence of Oxygen and Water.** MORITZ TRAUBE. (Vol. XV., p. 2,854.) Not only palladium containing hydrogen but also palladium which is free from hydrogen will oxidize carbonic oxide in the presence of water to

carbonic acid. Peroxide of hydrogen is also formed. Platinum behaves in the same manner. If platinum in the form of foil or wire is shaken with hydrogen, air and water a large quantity of peroxide of hydrogen is at once formed.

**Separation of Asparagin from Solution.** E. SCHULZE. (Vol. XV., p. 2,855.) Mercuric nitrate when added to a solution containing asparagin will precipitate it. The precipitate can be decomposed by means of sulphuretted hydrogen, again affording asparagin.

**Preparation of Indigo blue from Orthonitrobenzaldehyd.** AD. BAEYER and VIGGO DRAWSEN. (Vol. XV., p. 2,856.) If a solution of orthonitrobenzaldehyd in aceton is treated with water until a cloudiness appears, and subsequently with soda lye, baryta water or ammonia, the solution at first becomes yellow, then green, and after a time deposits large quantities of indigo blue.

**On Aromatic Antimony Compounds and a New Method of Forming Aromatic Arsenic Compounds.** A. MICHAELIS and A. REESE. (Vol. XV., p. 2,876.) When sodium is added to a mixture of cholride of arsenic, brombenzol and ether, chemical action at once ensues. On evaporation of the ethereal filtrate an oily residue remains which soon crystallizes. The product recrystallized once from hot alcohol is pure triphenylarsin. This heated with excess of chloride of arsenic in a closed tube for some time at 250° C. gives mono-phenyl-arsenious chloride :



The antimony compounds are formed in a similar manner. Antimonious chloride and brombenzol are dissolved in the appropriate proportions in benzol, an excess of sodium is added and all is heated for some time at the upright condenser. The filtrate upon evaporation and recrystallisation form a large quantity of alcohol, gives a pure stibin.

**On the Dopplerite of Aussee.** W. DEMEL. (Vol. XV. p. 2,961.) The author, after a series of quantitative analyses and tests to imitate the organic mineral dopplerite, concludes as the most probable, that the mineral is to be recognized as the calcium salt of one or more of the acids of the series of the humus substances.



**On Carbonic Acid Hydrate.** M. BALLO. (Vol. XV., p. 3,003. In testing a mineral water sold in commerce under the name *Salvator*, the author was struck by the action of the carbonic acid contained in the same. The water contains 2.35 grms. of free acid to the litre, and notwithstanding this large amount, the water does not sparkle at 8-10° R. It occurred to the author that in aqueous solution the carbonic acid might be present as hydrate. A proof for the existence of carbonic acid hydrate was given, when a metal was found which would dissolve in the carbonic acid solution accompanied by the evolution of hydrogen. Magnesium was found to answer the purpose.

**Methylating and Ethylating of Anilin and Toluidin.** H. REINHARAT and W. STAEDEL. (Vol. XVI., p. 29.) When the bromine or iodine hydrates are heated with the calculated amount of methyl alcohol and of ethyl alcohol to 145°-150° and 125° respectively, the secondary and tertiary bases are formed respectively. All the compounds are known except the mono- and diethyl-orthotoluidin.

**Action of Chlorocyanogen on the Potassium Compound of Pyrrol.** G. L. CIAMICIAN and M. DEMESTEDT. (Vol. XV., p. 64.) The authors obtained needle shaped crystals having the melting point 210°. The analyses gave figures corresponding to a formula  $C_4 H_4 N-CN$ . *i. e.*, cyanogen-pyrrol or tetrol-cyanamid. It is most probable, however, that the product is a polymerid of  $C_4 H_4 N_2$ , corresponding to the formula  $3 (C_4 H_4 N_2)$  and to the name tetrol-cyanuramid or tetrol-melamin. The behavior of this compound is similar to diphenyl-cyanamid, which latter was prepared by Weith (Berichte, Vol. VII., 843) from chlorocyanogen and diphenylamin.

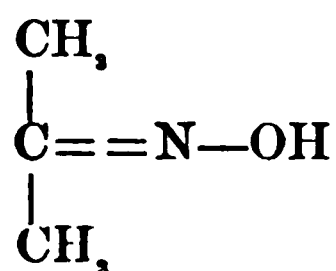
**On a New Method of Preparing Carbonic Oxide.** E. NOACK. (Vol. XVI., p. 75). The method is in brief as follows: A piece of combustion tubing is filled with zinc dust, leaving a channel for the passage of carbonic acid, which is generated in the ordinary manner. It is good to insert a bottle containing soda solution between the combustion tubing and the receiver. The carbonic oxide should be conducted through the bottle containing caustic soda. The zinc dust is not quite allowed to glow. With 200 grms. of zinc 20 litres of carbonic oxide were obtained; 13 litres of carbonic acid gave 11 litres of carbonic acid.



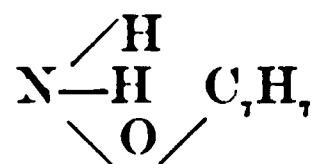
**Burning of Sulphur with White Phosphorescent Flame.** K. HEUMANN. (Vol XVI., p. 139). The author found that when sulphur is placed on a warmed plate in the dark a white phosphorescence is observed, entirely different from the blue flame occurring when sulphur is burned. Another method of observing the phenomenon is to heat a glass rod and then to dip it into powdered sulphur. If the rod has not been too hot the white phosphorescence is easily observed in the dark. A still better method is to heat the sulphur rapidly on a plate inside of a metallic air bath to 180°. In this manner the phosphorescence can be kept up for hours.

**On the Synthesis of Cinnamic Acid according to The Schwartz.** E. ERLÉNMEYER. (Vol. XVI., p.152). The author finds, after various attempts during a period of years, to synthetically prepare cinnamic acid, that phenyl-propionic acid and phenyl-propionic-acid are the only acids formed. Schwartz probably mistook phenyl-propionic-acid, which at the time of his publication was not known, for cinnamic acid.

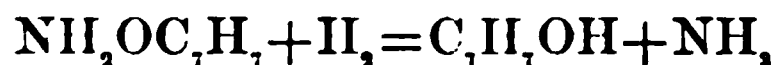
**On Isonitroso Compounds.** VICTOR MEYER. (Vol. XVI., p. 167. The acetoxine described previously by V. Meyer and Alois Tanney has the formula



Treated with acids it is decomposed into acetone and hydroxylamin. The base must hence have the formula



This can be proven by reducing the Benzyl-hydroxyl-amin with hydriodic acid, forming ammonia and iodobenzyl.

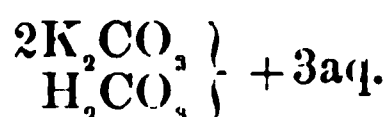


**On Paraxanthin, [a New Component of Human Urine.** G. SALOMON. (Vol. XVI. p. 195.) The method employed for obtaining the xanthin compounds was that given by Salkowski and Leube, "Die Lehre vom Harn" (Part I., p. 105). The last product, formerly supposed to be the xanthate only, and consisting of a silver salt, was decomposed by sulphuretted hydrogen. The remain-

ing phosphates and a small quantity of oxalate of lime were then removed, the solution slowly evaporated on a sand bath until the xanthin, which is soluble, with difficulty separated. This was then filtered off and from the filtrate the paraxanthin was obtained by evaporating to crystallization. The formula is most probably  $C_{15}H_{17}N_9O_4$ .

**Action of Chinolin on Chloroform and Iodoform.** O. O. RHOUSSOPoulos. (Vol. XVI., p. 202.) If chinolin is allowed to act on chloroform no reaction takes place even when the substances are heated together in a sealed tube to  $250^\circ$  for several days. At  $300^\circ$  the substances act partially only. If, however, iodoform is used, a reaction takes place easily at common temperature, forming a methan-trichinoil-ido-hydrate,  $CH(C_6H_7NI)_3$ .

**On the Sesquicarbonate of Potassium.** C. RAMMELSBERG (Vol. XVI., p. 273.) While the sesquicarbonate of sodium occurs as Troua (Ural) in the salt lakes of Africa and South America, no reference is found on the potassium salt. According to G. H. Bauer it has been found in a mineral water establishment of Dr. Struve & Holtmann in consequence of evaporation and crystallization of large quantities of bicarbonate solution by Dr. Lichtenstaedt. The crystals are not moist and do not decompose. They are—



Bauer's analysis (a).

Rammelsberg's analysis (b).

	(a)	(b)	Calculated.
{ Potash . . . . .	46.54	46.59	47.96
{ Carbonic acid . . . . .	21.79	21.80	22.39
Carbonic acid . . . . .	11.54	12.22	11.20
Water . . . . .			18.45
			<hr/> 100.00

**On Oenanthalanilin, Oenanthalxylidin and Oenanthalnaphthylamin.** A. R. LEEDS. (Vol. XVI., p. 287.) 70 grms. oenanthal and 57 grms. of anilin were mixed gradually. The temperature greatly rises (from  $27^\circ$  to  $89^\circ$ ).

70 grms. of oenanthal and 74 grms. of xylidin were mixed for preparation of oenanthalxylidin.

70 grms. of oenanthal were added to 88 grms. of naphthylamin, in consequence of which the latter rapidly melted a liquid having been formed of the temperature  $75^\circ$ . All three compounds were

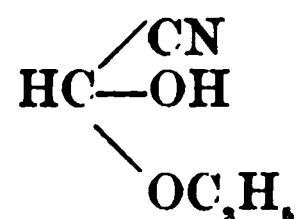
heated for six hours at the upright condenser. The best method for purification was found to be as follows: Each of the three compounds was dissolved in about 150 grms. of glacial acetic acid and heated for several hours on the water bath. Then water was added in large excess which precipitated, the oenanthal, anilin-xylidin-naphthylamin respectively leaving the acetates in solution. The analyses of all three corresponded to the formulæ: Oenanthalanilin  $C_8H_7NC_7H_{14}O$ ; Oenanthalxylidin  $C_8H_{11}NC_{17}H_{14}O$ ; and Oenanthalnaphthylamin  $C_{10}H_9NC_7H_{14}O$ .

**On Cryptidin.** A. R. LEEDS. (Vol. XVI., p. 289.) An oil was obtained by distilling 155 grms. of xyloidinacrolein in portions of 20 grms. No note was taken of the temperature of distillation until it had reached  $360^\circ$ , 11 grms. or about 7% of the oil were obtained. The oil has a disagreeable odor and a very bitter taste. It forms crystalline salts with sulphuric, hydrochloric and other acids. The best method of purification is to decompose the hydrochloric acid salt with alkali. The purified and dried oil has the boiling point  $270^\circ$ . It has a reddish color. The analysis corresponded to the formula of cryptidin:  $C_{11}H_{11}N$ .

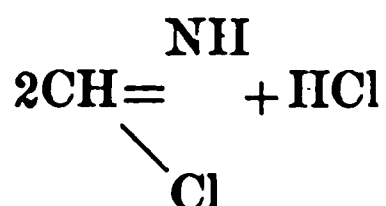
**On the Action of Phtalic Acid-Anhydride on Chinolin.** M. C. TRAUB. (Vol. XVI., p. 297.) As shown by O. Fischer it is easy to extend the phtaleïn reaction on dimethylanilin as a confirmation of the similar action of phenols and tertiary aromatic amines. The author endeavored to further extend these reactions to tertiary aromatic amines, the nitrogen of which has entirely different combining conditions from that of dimethylanilin, mainly to pyridin and chinolin bases. When phtalic acid anhydride is treated with chinolin 3 to 4 hours at  $150^\circ$  a reaction takes place. After removal of excessive chinolin a sticky mass remains, which is purified by glacial acetic acid and crystallized from benzol. The analysis shows that a reaction similar to the anthrachinon formation has taken place. The formula being  $C_{17}H_9NO_2$ , the compound is termed chinophthalon by the author.

**On the Combinations of Hydrocyanic Acid with Hydrochloric and Hydrobromic Acids.** L. CLAISEN and F. MATTHEWS. (Vol. XVI., p. 308.) Some time ago it was observed by Pinner and Klein, that when hydrochloric acid was passed into an alcoholic solution of hydrocyanic acid diethyl-glyoxylic acid was formed besides other products. Claisen and Matthews supposed that the reaction took place as follows: formic ether is at first

formed, and this in the same manner as the aldehyds combined with the hydrocyanic acid to a nitrite.



and that this was converted into the glyoxylic acid derivative by the action of the alcohol and the hydrochloric acid. The authors could not prepare such a product from formic ether and hydrocyanic acid, but found that hydrochloric acid is greedily absorbed by the mixture, giving rise to a double compound of hydrocyanic and hydrochloric acids.  $2\text{CNH} + 3\text{HCl}$  is the resulting compound. The formic ether plays no part, as the reaction takes place in presence of any other ether (benzoic ether, acetic ether.) The reaction takes place at as low a temperature as  $15^\circ$ . The constitution of this compound is probably best expressed thus :



The hydrobromic acid compound is similarly obtained.

Hydrocyanic acid and fuming hydrochloric acid were mixed in the proportions, so that for one mol. of hydrocyanic acid, exactly one mol. of water was present. After proper treatment, an oil with boiling point,  $190-210^\circ$ , is obtained, which, according to a nitrogen determination, was found to be formamid. Contrary to previous statements, hydrocyanic acid can be converted into the corresponding amid by careful treatment with hydrochloric acid.

### Formation of Arsenides by Pressure. W. SPRING. (Vol XVI., p. 324.)

The author publishes a series of experiments and results obtained by pressing pulverized arsenic separately with various metals.

**Zinc arsenide.**—A mixture of zinc filings and pulv.-arsenic, in the proportions expressed by the formula  $\text{Zn}_3\text{As}_2$ , was subjected to a pressure of 6,500 atmospheres. The resulting block was again filed and repressed. A homogeneous bright metallic mass was obtained. The elevation of temperature plays no part, being but very slight; hence zinc and arsenic combine by mechanical energy alone.

**Lead arsenide.**—After two pressures of the mixed filings, the

mixture corresponding to the formula  $\text{Pb}_3\text{As}_2$ , a homogeneous block of metallic lustre was obtained. It is hard and brittle.

Tin arsenide.—This is very easily formed, even when mixed in different proportions. When mixed in the proportions corresponding to  $\text{Sn}_3\text{As}_2$ , a white metallic mass is obtained, brittle, and melting with more difficulty than tin.

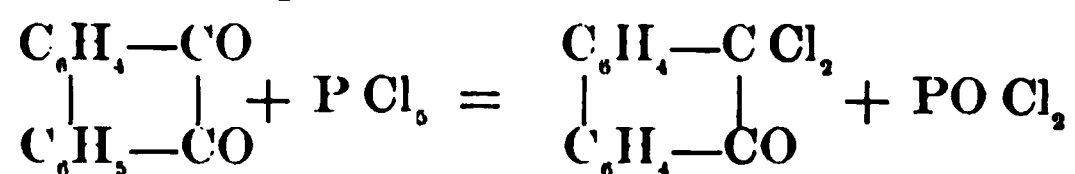
Cadmium arsenide.—After three pressings the combination was complete. From experiments the author finds that by pressure a better alloy can be obtained than by melting the metals together. This is probably because the dissociative tension of the arsenides is so great as to prevent a good combination.

Copper arsenide.—This is not as easily formed as the preceding alloys. After a first pressing of a mixture corresponding to the formula,  $\text{Cu}_3\text{As}_2$ , all particles of copper and arsenic can be found separately. After about eight pressings the mass becomes homogeneous of metallic lustre, brittle and fine grained. The mass has a whitish-gray lustre.

Silver arsenide.—This is as difficult to form as the copper arsenide. The mass becomes homogeneous, very brittle, has a metallic lustre, and a gray-bluish color.

Arsenic alone in the amorphous state when subjected to 6500 atmospheres pressure, changes its appearance and becomes metallic on the surface. About one-fourth is converted into crystalline form

**On the Action of Phosphorus Chlorides on Phenanthrenchinon.** B. LACHOWICZ. (Vol. XVI., p. 330.) Phosphorus pentachloride and phenanthrenchinon react upon each other vigorously when slightly warmed and a crystalline mass is formed. It is purified by crystallization from chloroform and dried over sulphuric acid. The formula to which the analysis corresponds is  $\text{C}_{14}\text{H}_8\text{OCl}_2$ . The reaction takes place as follows :



The author terms this compound phenanthrendichlor-keton. Its melting point is  $165^\circ$ .

**On Allyl-succinic Acid and a Carbo-caprolactonic Acid.** EDW. HJELT. (Vol. XVI., p. 333.) The allyl-succinic acid was obtained synthetically from malonic-acid-ether. By action of sodium ethylate and chloro-acetic ether on malonic-acid-ether the ethenyltricarboxylic-acid-ether was produced and the allyl group

was introduced into this. The allyl-ethenyl-tricarmonic-acid-ether boils between  $280^{\circ}$  and  $290^{\circ}$ . From the barium salt the acid was obtained pure. The formula according to analyses is  $C_8 H_{10} O_6$ . When treated with hydrobromic acid this acid gives a crystallized acid, which melts at  $159^{\circ}$ , at  $160^{\circ}$  this loses carbonic acid. The residue allyl-succinic acid solidifies and is obtained pure by crystallization from alcohol. Its formula is  $C_7 H_{10} O_4$  and is isomeric with teraconic acid. When heated to  $250^{\circ}$ , water is at first separated and oil distills over, which in contact with water again forms allyl-succinic acid. The acid dissolves in forming hydrobromic acid. By treating with water and extracting with ether, and acid oil, free from bromine is obtained, which crystallizes in vacuo. This acid was found to be the lactonic acid, of the formula  $C_7 H_{10} O_4$ .

# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Regular Meeting May 4, 1883.*

At Mr. Stebbins' request Prof. Leeds occupied the chair.

The minutes of the February meeting were read and approved.

The minutes of the meetings of the Board of Directors, held January 16, 1883, and March 20, 1883, were also read.

The following papers were read :

1. On apparatus for producing high vacuum with low pressure, by C. J. Lawler.

2. Platinic Iodide as a test reagent for deleterious organic substances in potable waters, by Dr. A. R. Leeds.

3. A note by Dr. Breneman, on a "Eudiometer for Rapid Gas Analysis."

4. A note by Prof. A. R. Leeds, on "The Conversion of Carbon Monoxide to Dioxide by Active (*i. e.* Nascent) Oxygen."

The papers were followed by discussion participated in by Messrs. Stebbins, Casamajor, Alsberg and Leeds, after which the Society adjourned.

THOMAS S. GLADDING,  
Recording Secretary.

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## NEW ASPIRATOR, CAPABLE OF GIVING A HIGH VACUUM WITH A LOW WATER PRESSURE.

BY C. J. LAWLER.

In the London *Engineer* for 1876, Mr. Bronson has described an aspirator, giving results which are about the same as those obtained with the apparatus which I propose to describe. The only merit claimed for the new aspirator is its greater simplicity.

With the new apparatus a vacuum equal to the barometer vacuum may be obtained, minus the tension of the water vapor corresponding to the temperature of the water used to work the apparatus.

This result can be reached with a water pressure not exceeding  $5\frac{1}{2}$  pounds.

The good results obtained with such very low water pressure, by means of an apparatus of very simple construction, have been

received with great favor by several friends, who have urged that the publication of the results obtained would be useful and interesting to other chemists.

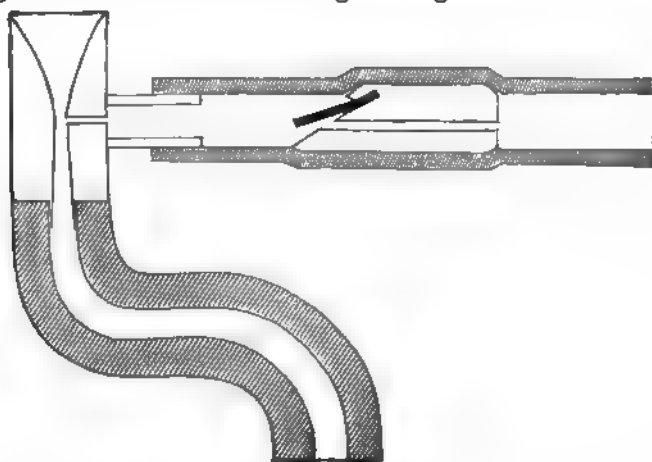
Experience has shown that a high vacuum is not necessary for filtration, but for other purposes, such as fractional distillation, a high vacuum is a great help.

High water pressure cannot be obtained in many laboratories, especially those situated in the lower part of New York city, and chemists constantly complain that they are unable to secure a vacuum with their filter pumps.

In order to get an aspirator which gives the best results with lowest water pressure, it is simply necessary to adapt a few well-known principles in Hydrodynamics to the end in view.

A volume of water thirty-four feet high will support a column of mercury thirty inches in height. In order to do the same amount of work with a less "head" of water, a greater volume of the liquid must be employed. As a mass falling from a given height exerts a definite amount of energy in its fall, to secure the same amount of energy from a body falling from a lesser height, the mass must be increased, *i. e.*, the mass must be inversely proportionate to the height.

We must also consider that the form of tube or nozzle which has the greatest co-efficient of discharge must give the best results.



This shape, as shown by Venturi, is a tube whose interior presents the appearance of the frustrum of a cone, the sides of which diverge at an an angle of five and sixteen one hundredths degrees,



and whose length is nine times that of its lesser base. The discharge in this case is 2.5 times greater than through a thin plate, 1.9 times greater than through a short cylindrical tube, and 1.46 times greater than the theoretic discharge (Haswell).

The water enters the aspirator through a short funnel, whose sides converge towards the narrow part of the cone. The aspirating hole is at the junction of the cone and funnel, and should not be more than one-third the diameter of the throat of the apparatus.

Conical diverging arteries give their best results when submerged, but, as this is not convenient in the case of aspirators, the same end may be attained by attaching to the extremity of the pump a short piece of lead-tubing of slightly larger calibre than the discharge, bent at least twice at right angles.

The efficiency of this pump is shown by the following comparison with that of Prof. Richards (Chem. News, 1876, pp. 141.) The barometer standing at 29.8 inches, temperature of water in hydrant 47° Fah.

Water pressure.	New pump.		Richards' pump.	
1 pound	4.8	inches Mercury	1.0	inches Mercury
2 "	10.7	" "	1.6	" "
3 "	17.8	" "	2.3	" "
4 "	24.7	" "	2.8	" "
5 "	29.2	" "	3.4	" "
5½ "	29.4		3.8	" "
25 "			29.2	" "

No disparagement is intended in this comparison to Prof. Richards' aspirator, which was designed for high pressures, and which works admirably with a pressure of twenty-five pounds.

While it was an easy matter to get the necessary data for the construction of the aspirator, it was a matter of no little difficulty to get a workman that would rigidly adhere to these data. Fortunately I found in Mr. Ketchum, 171 Gates Avenue, Brooklyn, a person qualified for the work. To this gentleman I am also indebted for designing an ingenious and efficient check valve, which is a necessary adjunct to all aspirators which work with water-pressure. His method of setting the valve is also very ingenious. A piece of brass rod, one-quarter inch in diameter, and one-quarter inch long, is bored, turned and filed to the shape shown in cut. A small piece of thin sheet rubber, one-sixteenth inch smaller than the face of the valve, is permanently fixed to its seat by a lug cut in the upper

part of the face, this is placed in the end of the piece of rubber tubing which is attached to the aspirating tube, by first introducing a thin metal tube, whose internal diameter is slightly larger than the diameter of the valve. The latter is then dropped into the tube, and by means of a small rod, pushed into place just beyond the metal tube which is then withdrawn. The rubber tube embraces the valve so securely that it is impossible to displace or disarrange it. This method is superior to the older one of cementing the valve in the aspirating tube.

A pump actuated by water-pressure has some advantages over the Bunsen Pump which are worthy of attention. It is less expensive, less difficult to place in position, the degree of vacuum is easily regulated by turning on just sufficient water to secure the desired result, avoiding pinch-cocks which injure the rubber tubing, it is not liable to freeze, because there is no great length of pipe to be exposed, an attachment for producing a blast can be added with less trouble and expense.

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## PLATINIC IODIDE AS A TEST-REAGENT FOR DELETERIOUS ORGANIC SUBSTANCES IN POTABLE WATERS.

BY ALBERT R. LEEDS.

In the *Chemical News* 43, p. 180, M. Field has proposed to employ the reaction of certain organic substances upon a very dilute solution of platinum iodide in solution of iodide of potassium, and by their effect in the removal of the rose-color of this solution, to determine the fact of their presence in greater or less amount. Although the author proposes to employ the test only, or chiefly to establish the fact of the freedom of the water under examination from sewage contamination and from albuminous matters, yet the establishment of this fact alone is of the highest importance. The introduction of specific tests for the various kinds of organic matter, fresh or partly decomposed, which might possibly be present in potable water, would mark an advance in the practice of water analysis, and any proposition looking in this direction merits careful study.

After verifying Field's statements as to the decolorising effect of

certain of the most important organic substances upon platinum iodide solution, I compared the results arrived at by the use of platinum iodide and those obtained by the customary methods of water analysis. Moreover, regarding it probable that the platinum salt would undergo reduction in presence of organic matter when exposed to sun-light, I exposed the tests in a duplicate series of determinations to the action of sun-light, and found my anticipation confirmed. The experiments were made upon a series of samples, the other constituents in which had been determined with exceeding care. In the first series a test solution was made of the same strength as that employed by Field, or one part of platinic chloride in 500,000 parts of a very dilute solution of potassium iodide; 50 c.c. of this solution was added to 100 c.c. of the natural water under examination, and allowed to stand in diffused light for 24 hours. In the second series, 1 part of platinic iodide was dissolved in 200,000 parts of a dilute solution of potassium iodide, and 100 c.c. of this solution added to 100 c.c. of the samples of water, and the solutions exposed in wide comparison-tubes for 48 hours to the sun-light. The results were as follows:—

		In diffused light	In sun-light
Sample No.	227*	Very faint pink	Light rose
"	230	Pink just visible	Pink
"	231	No color	Smoky pink
"	234	Decided pink	Rose
"	235	Faint pink	Rose
"	228		Dark rose
"	232		Faint pink
		with deposit of platinum	
"	233		Smoky pink

It will be noted that in the second series, samples 231, 233 and 232 were so far reduced that a grayish deposit of platinum was formed on the walls of the tubes. In the last, this deposit was tested first with concentrated nitric acid, in which it would not dissolve, and afterwards with aqua regia, in which, after heating, it dissolved to a deeply-colored yellow solution. Since the 100 c.c. of platinic iodide solution was nearly decolorised, the amount of metallic plati-

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\* These laboratory numbers refer to a series of samples taken from the Schuylkill River in the month of January, 1883, when the water-supply of Philadelphia became so nauseous that I was requested by the water department of that city to investigate the causes of its temporary non-potability.

num precipitated amounted to nearly 5 mgrms. In other words, in badly polluted waters the reduction of the platinum salt in sun-light might be employed as a means for estimating the relative amounts of those organic bodies which are capable of effecting such a reduction in sun-light. It was noted, however, that 100 c.c. of a solution of 1 part of platinum iodide in 500,000 parts of a very dilute solution of potassium iodide in *distilled* water became perceptibly lighter in tint when exposed along with the others for the same period of 48 hours. Inasmuch as the solutions were somewhat warmed by the sun-light, and heat has some effect in the removal of the color, this disturbing element would have to be guarded against.

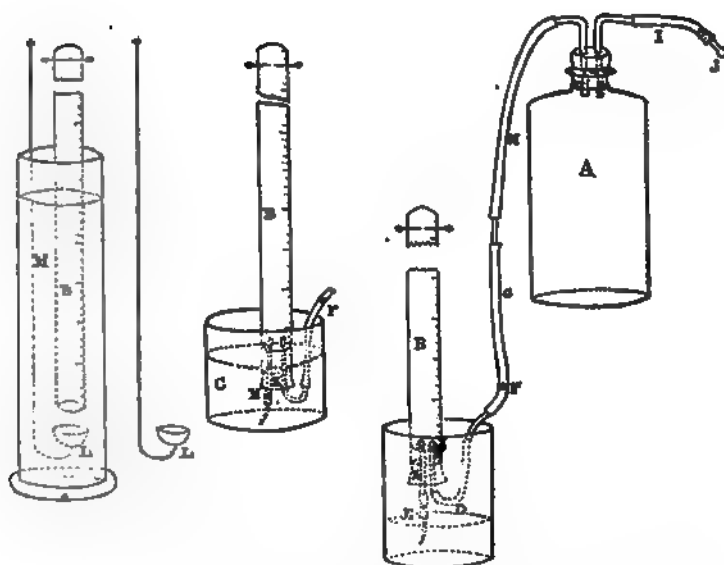
Since this time, an actinic method of determining the putrescible organic matter in potable waters has been regularly employed, and proven so satisfactory, that further experiments with platinic iodide in this direction have been abandoned. But the new ground broken by Field in the domain of water-analysis, deserves to be sedulously cultivated, and the search for specific tests for particular contaminating substances should be carefully followed up.

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## ON AN APPARATUS FOR RAPID ANALYSIS OF GASES.

BY A. A. BRENNEMAN, S. B.

The apparatus here described is designed for rapid analysis of gaseous mixtures where only a moderate degree of accuracy is required. It is possible nevertheless to secure very good results with it, if it be carefully handled and if its known source of error be guarded against. As an example of the results that may be obtained with it, two analyses of air may be quoted which yielded respectively 21.04% and 21.13% by volume of oxygen instead of 20.96% which is probably a fair average for the most accurate analyses. The oxygen was absorbed by a solution of pyrogallie acid in caustic potash, in the proportion of 2 grms of the solid acid to 5 c.c. of a solution of  $\text{KHO}$  (sticks) in twice its weight of water and 10 c.c. of water were further added to the solution. The absorption of oxygen in each case, occupied about 12 minutes. The excess of oxygen indicated is probably to be accounted for by the solution of part of the gaseous mixtures in the water used for rinsing, but the error belongs to all absorption-methods of analysis, where the gas is measured over water.



The apparatus and its use will be readily understood from the accompanying diagram. A is a bottle holding a liter or more. It is closed by a soft rubber stopper carrying two tubes, one of which communicates with a water air-pump, the other with the gas burette. After exhausting the bottle at the pump, the tube I is closed by the stopper is already closed by a bit of stirring rod, plugging the rubber tube G at F and serving also as a valve. It is opened by simply pinching up a portion of the rubber tube about it, thus forming a channel around the plug. The gas burette B is closed by a rubber stopper K, carrying two tubes, one of which connects with A, the other carries a valve at E and a pointed tube of glass at its end.

The gas burette is filled with gas over the pneumatic trough C, the cork K being removed. It is then transferred by means of the deep spoon L to the measuring cylinder, the volume of the gas is noted with the existing temperature, the burette is again brought to the trough, K is inserted and then by opening the valve F the level of water in the burette is brought to the inner surface of the cork K, the water passing into the exhausted bottle L. This bottle is used in the same way, subsequently, to receive all drainings and rinsing water from the burette.

The absorbing liquids are introduced by bringing the point of the tube below E into the liquid and opening E; liquid rushes into the partially exhausted tube to replace the water previously withdrawn.

The burette is shaken to distribute the liquid over its sides and more of the liquid is introduced as the absorption proceeds. When absorption is complete the liquid is withdrawn as before, the tube is rinsed with successive small quantities of water, and the gas finally measured again over water in the cylinder M.

The obvious errors of the apparatus are the entrance of air about the cork K, and separation of gases from the liquids used in the burette when the level of the liquid in the latter is reduced. A soft, well fitting rubber stopper will remove the first objection and as an additional precaution the mouth of the burette is kept below the surface of the water whenever it is not necessary to manipulate the burette. In practice the entrance of external air through leakage is inappreciable during the time of the analysis.

The second source of error is avoided by using such quantities of absorbing liquids that the diminution of pressure within the burette may be slight. During the time of greatest exhaustion, that is during the withdrawal of liquids, the current tends to carry minute bubbles that may separate downwards and into the bottle, and with a good vacuum (20-25 inches of mercury) in the latter, the time is too short to permit of any important volume of gas separating from the liquid.

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## THE CONVERSION OF CARBON MONOXIDE TO CARBON DIOXIDE BY ACTIVE (*i. e.* NASCENT) OXYGEN.

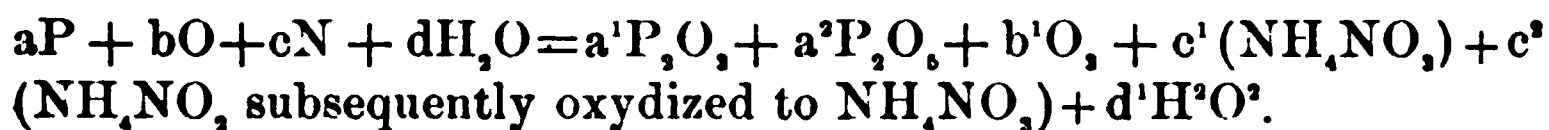
BY DR. ALBERT R. LEEDS.

In the early part of the year 1879, I was led by certain theoretical considerations to regard it as eminently probable that carbonic oxide would be converted by nascent oxygen into the highest stage of oxidation, and that by properly devising the nature of the experiments, and adequate precautions in the execution of their details, the direct oxidation of carbon monoxide to dioxide by nascent oxygen at ordinary temperatures could be demonstrated. Repeated trials amply justified these anticipations. The final experiment, some account of which will be found in the Jour. Amer. Chem. Soc., I, 232, was performed as follows: A 5-liter jar, containing 5 sticks of phosphorus and a little water, was filled with 2500 cc. of carbonic oxide, and the remaining space with air. The carbonic

oxide was generated from potassium ferrocyanide and sulphuric acid, and washed with water, caustic potassa, and barium hydrate. On transferring it to the jar, it was again washed through a similar train of purifiers, a final V-tube filled with barium hydrate solution remaining pellucid, proving absence of any trace of carbon dioxide. The air was purified in the same manner. At the expiration of 18 hours, the gases remaining above the moist phosphorus in the jar were drawn, first through a moist cotton filter, then through a potassium iodide solution, and finally through two baryta waters. The faint rose coloration which the iodide solution struck with starch water, was found on titration to correspond to 0.04 mgrms. ozone in the total volume of gas drawn over. The last baryta was absolutely pellucid, the first had absorbed 14 mgrms. carbonic acid. The carbonic acid estimation was made by decomposing the barium salt, and weighing the carbonic acid evolved, for fear that some traces of oxides of phosphorus might have escaped from the purifiers, and entered into combination with the baryta.

Later on, further details as to precautions employed in these experiments will be given, in connection with the adverse criticisms to which they have been recently subjected.

Somewhat later in the same year, a related field of experiment was entered upon. This was to determine conclusively, whether or no the triatomic molecule of oxygen was capable of effecting what the individual atom of oxygen, at the moment of dissociation of the molecule of oxygen, had been demonstrated to be capable of doing. It should be premised, that the decompositions and recombinations which occur when phosphorus is oxydized at the expense of, and with simultaneous reduction of the oxygen molecule, are complicated in their nature. An operose determination of the factors of this reaction (Jour. Am. Chem. Soc., I, 146 and II, 37), has shown that it may be represented by the equation



The actual values of the coefficients would depend, as was shown in another place, upon the temperature at which the reduction of the oxygen molecule is effected, and the values, a, b, c, d, of the members of the equation. But the important point established was that ozone, hydrogen peroxide, and nitro compounds of ammonia, were always formed, and their quantities always bore a certain relation to one another. Moreover, that the explanation of these

facts could be found only in the supposition of the formation of active oxygen, and the subsequent generation of ozone was no more an essential feature of the reaction than the contemporaneous and invariable production of hydrogen peroxide, and ammonium nitrite and nitrate.

In some earlier experiments, which had been performed with great care by Profs. Remsen and Southworth,\* the conclusion had been arrived at that carbonic monoxide was not oxidized to carbon dioxide by ozone.

As above stated, I was induced to renew the investigation,† inasmuch as certain preliminary experiments, which I had performed, had shown that carbonic oxide might undergo conversion into carbonic acid, under the same circumstances which brought about the oxidation of the oxygen molecule to the state of ozone. These were notably two : 1st. The oxidation of carbonic oxide by nascent oxygen, evolved in an atmosphere of moist air over phosphorus, as detailed above. 2d. The simultaneous formation of carbonic acid and ozone in a mixture of carbonic oxide and oxygen, subjected to the influence of the silent electrical discharge. Without pausing to rehearse *in extenso* the experiments by which this latter point was established, it will suffice to state that a quantitative determination resulted in showing that after  $1\frac{1}{2}$  liters of carbonic oxide had been submitted to the action of the silent discharge, in company with oxygen, 0.0271 grms. (misprinted 0.0271 mgrms. loc. cit.) of carbon dioxide had been formed.

Moreover, I had been enabled, by the use of a modified form of Siemens' ozonizer, to make use of large volumes of oxygen, raised to a higher degree of ozonation than the ozonized gas hitherto employed, and I thought that the theoretic importance of the subject warranted the institution of a fresh attempt to discover whether, with ozone of this degree of concentration, no oxidation of carbon monoxide occurred.

The actual experiment was conducted as follows :—

Oxygen, previously freed from every trace of carbon dioxide, was ozonized by passage through an ozonizing battery to the extent of 72 mgrns. of ozone per liter. It was then passed in excess, together with carbon monoxide likewise completely purified, through a tube one meter in length, which was placed *beyond* the ozonizer. But although the current of mingled ozonized oxygen

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\* Am. Jour. Sci., III, 11, 136.

† Jour. Amer. Chem. Soc., I, 450.



and carbon monoxide was made to flow with a very slow current, and though the experiment was continued for many hours, the baryta water through which the mixed gases finally made their escape, remained entirely pellucid. In other words, oxygen containing a very large percentage of free ozone, is not capable of effecting the oxidation of carbon monoxide.

Commenting upon this result, the inference was drawn:—"In this case, the energy of combination of the two bonds, presumably free, of the carbon monoxide molecule, appears to be of less valency than the energy with which any one atom in the ozone molecule, is retained by the other two. In the two former instances" (the oxidation of carbon monoxide by nascent oxygen, and the generation of carbon dioxide in a mixture of carbon monoxide and oxygen submitted to the silent discharge) "the ozone molecules are in the process of formation, and in the pre-existent stage (in which the oxygen molecule undergoes dissociation,) the liberated oxygen atom is seized upon by the unsaturated carbonic oxide."

Subsequently\* these views were still further generalized in an essay which had for its object the quantitative investigation of the various factors in the complicated reactions connected with the oxidation of phosphorus in moist air. It had previously been shown† that the amount of ozone produced by the oxidation of phosphorus under given circumstances, is constant for any particular temperature, and consequently, the generation of ozone may be graphically represented by a curve, the production at 6° C., being nil, rising to a maximum at 24°-25° C., and then rapidly diminishing as the temperature approaches 44° the melting point of phosphorus. As additional proof of the constancy of the phenomena occurring in the oxidation of phosphorus in moist air, it was established in the progress of this essay, that the amounts of ozone and hydrogen peroxide bear a constant relation to one another and to the air passed over. A similar proposition holds true of the phosphoric and phosphorous acids, and of the ammonia and nitric acid—the amounts of the latter bodies being in the ratio requisite to form ammonium nitrate. Later on it was shown that, neglecting the small amounts of hydrogen peroxide, which were held back by the wash-waters through which the escaping gases pass, the ratio of the hydrogen peroxide generated, was to that of the ozone as something over one to three, under the conditions of that particular experiment.

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\* Jour. Am. Chem. Soc. II., 34.

† Jour. Am. Chem. Soc. I., 8.

These various results caused me again to reiterate the wide generalization which had been brought forward in the earlier articles concerning the production of ozone under the conditions studied :—that it was connected with the uneven quantivalences of the elements taking part in the reaction. “If this hypothesis be true, then we should anticipate the development of ozone whenever oxidation of a perissad occurred at temperatures compatible with the stability of the ozone molecule. Even at the temperature of combustion of hydrogen, this is supposed by C. Than to be the case. He explains in this manner the presence of the ozone, which he states he has detected in the combustion of hydrogenous substances generally, and its absence in the combustion of carbon.”

“In entering into new combinations, the oxygen molecules must undergo temporary resolution into their constituent atoms. These while *en route* to take up new positions in other combinations, and animated by their atomic energy, or energy of the nascent state, may either oxidize the oxygen molecule, or the nitrogen molecule, or the molecule of water. In the first place, ozone would be produced ; in the second, regarding water as the basic body and NNO as the nitryl, there might be formed, as Hunt has indicated, ammonium nitrate ; in the third, hydrogen peroxide.”

This recapitulation is here given in order to recall the gradual evolution of the hypothesis of the necessary existence of active or nascent oxygen, as a body distinct from ozone, in order to explain observed phenomena. According to these views, the oxidizing effect of palladium-hydrogen, as noted by Hoppe-Seyler, at a later period in the history of these researches,\* was a particular case of the oxidation of a perissad (in this case hydrogen) at a temperature especially compatible with the stability of the ozone molecule. The intervention of nascent hydrogen, or active hydrogen, is not needed to explain the production of hydrogen peroxide when hydrogenium or palladium-hydrogen comes in contact with water. Hydrogenium is a perissad metal endowed with the most eminent deoxidating or reducing powers. When it reduces the molecule of oxygen, a temporary resolution of the molecule into its constituent atoms must necessarily take place. In other words, hydrogenium stands in the same relation to the production of hydrogen peroxide, as phosphorus does when oxidized in presence of moist air. Like the phosphorus it directly reduces the oxygen molecule, and sets free the oxygen

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\* Activation of oxygen by nascent hydrogen. Ber. der deutsch Chem. Gesell., XII, 1551.

atom. This active oxygen so formed, is the efficient cause of all the subsequent phenomena. It starts the series of chemical changes, and is the principal and all important factor, as related to which and resulting from which, the generation of the ozone, hydrogen peroxide, and ammonium nitrate and nitrite, are three correlative and necessarily dependent, but inevitably and indissolubly connected, consequences. According to this hypothesis, the generation of ozone is not the central fact around which the possible formation of active oxygen and the other phenomena are grouped, but it is a secondary phenomenon and is of no more importance so far as the theory of the reaction is concerned, than the simultaneous development of hydrogen peroxide, and, in presence of nitrogen, of nitrogen compounds.

The foregoing history of previous investigations is given, in order to render intelligible the attitude in which I stood with regard to the statements recently made by Profs. J. Remsen and E. H. Keiser in a paper "On the Conduct of Moist Phosphorus and Air towards Carbon Monoxide" (Amer. Chem. Jour. IV, 454). It will be further necessary only to state that my views are not the same as those advanced by Hoppe-Seyler, as may be seen from the different induction which I drew from Hoppe-Seyler's own experiments. For in his original experiments this investigator did not suspect the necessary presence of peroxide of hydrogen, as an indispensable factor in the reactions observed. Subsequently (Ber. der deutsch Chem. Gesell., XIV, 976) I pointed out that this must be the case, and on performing the experiment, quantitatively determined the amount of peroxide formed. The only reasons for previously anticipating, and actually performing the experiments confirmatory of the presence of hydrogen peroxide, were the theoretical considerations connected with the development of active oxygen above enunciated. Ignoring these results, M. Traube in a later issue of the *Berichte* (XV, p. 659) brought forward the formation of hydrogen peroxide under these circumstances, as his own observation, and has founded upon this production of hydrogen peroxide a hypothesis concerning the validity of which, and concerning the experimental verification of the experiments upon which it is based, I shall defer writing until the publication of a subsequent article.

After recounting certain preliminary experiments, the crucial one, by which Remsen and Keiser hold that they have conclusively established the non-conversion of carbon monoxide to dioxide in the presence of moist air and phosphorus, and so far as the evidence

afforded by this experiment is concerned, the non-existence of active oxygen, is stated (by them *loc. cit.*), as follows:

“An apparatus was finally constructed in which these were entirely excluded, or, at least, so protected that the ozone could not possibly reach them. Although a number of different forms of apparatus were tried, we need only describe that which we finally decided upon as the best. This consisted of a flask of from three to four liters capacity, provided with a doubly perforated cork stopper. Through this there passed one glass tube reaching to the bottom of the flask, and another reaching only half way. Outside the flask the shorter tube was connected with the apparatus intended to remove the carbon dioxide from the air, and the longer tube was bent twice at right angles, and then passed through the stopper of a V-tube about eight inches high. Between this V-tube and a second like it, connection was made by means of a doubly bent glass tube. In the flask there were placed usually two or three sticks of phosphorous, each three or four inches long, and enough pure water to somewhat more than fill the neck when the flask was inverted. The V-tubes were filled with ignited asbestos, and there was then added some mercury, so that when the tubes were inverted, in which position the entire apparatus was placed when in use, the metal covered the corks with a layer from three-quarters of an inch to an inch in thickness. The connecting tubes passed, of course, in each case through the layer of mercury. The vessel containing the clear baryta water was also connected with the last V-tube by means of a mercury joint similar to those above described. The baryta water was protected from the action of the air by placing before it a small V-tube containing potassium hydroxide, and this last tube was connected with an aspirator. Before connecting the bulbs containing the baryta water, air freed from carbon dioxide was drawn slowly\* through the apparatus. On now connecting with the baryta water bulbs no precipitate was formed. Even on allowing the air to remain in contact with the moist phosphorus for periods varying from one to twelve hours, no carbon dioxide could be detected. This experiment was tried over and over again at different temperatures, but always with the same result.

About one-third of the air in the ozone flask was now replaced by carbon monoxide, from which all dioxide had been scrupulously re-

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\* This operation requires care and constant attention. The phosphorus not unfrequently takes fire. In one case in our experience it took fire after having stood quietly and unmolested for several hours.

moved. The mixture was allowed to stand for some hours and then drawn through the baryta-water bulbs, when no precipitate was formed. This experiment was repeated a number of times with the same result. In some cases the air and carbon-monoxide were drawn together slowly for a long time over the moist phosphorus, but this made no difference in the result. So frequently was the experiment performed, so great were the precautions against error, and so undeniably negative were the results, that, in spite of the explicit statements of Leeds and of Baumann, we do not hesitate to say that carbon-monoxide is not oxidized when exposed to the action of air and moist phosphorus. It follows, of course, from this, that the action of air and moist phosphorus on carbon-monoxide furnishes no evidence in favor of the view that there is an active condition of oxygen distinct from ozone."

The authors further say : "Leeds' error is easily explained. In the first place, he took no special precautions to protect the corks from the action of the ozone, and in the second place he passed the gases through a filter of moist cotton for the purpose of removing 'Oxides of phosphorus.' Now cotton, as it is usually met with, yields carbon-dioxide when subjected to the influence of ozone, so that the carbon-dioxide noticed by Leeds was probably the result of the action of ozone on the cotton or the oily substances which always adhere to it."

With regard to this explanation, it may be said that at a very early stage in experimenting with ozone, the observer is compelled to note the destructive action of ozone on corks and rubber connections, and either to eliminate them altogether or to boil the corks in paraffine and cover them with the same, both of which last precautions were taken in the present instance. The action of the very attenuated form of ozone obtained from phosphorus is excessively slight, as I knew when speaking of its employment in connecting ozone apparatus at an earlier date (J. Am. Chem. Soc. 11, 150) "Paraffine does not perfectly withstand the action of ozone, being slowly decomposed with the formation of carbonic acid. In the course of long continued use, the paraffine joints employed by the author in connecting the ozonizing elements of his large electrical ozonizer (ib. 1, 440) were destroyed, and had to be renewed from time to time. The amount of action on the joints was progressive, the paraffine connection between the first and second ozonizing element requiring to be changed very seldom, while that between the eleventh and twelfth elements needed frequent renewal." \* \* \* \*

“In experimenting with ozonized air, containing so small an amount of ozone as that employed in the investigation described in the present article” (ozonation by phosphorus), “the connections may be made of paraffine without entailing serious error, but in researches of greater nicety, where not merely the relative but the absolute amounts of ozone have to be determined, paraffine connections cannot be employed.”

The cotton employed to remove the oxides of phosphorus, and I should have added in this as I have in other places, that cloud of vesiculated peroxide of hydrogen (the antozone of Schönbein and other authors) which by its passage through *water*, etc., without absorption, so puzzled earlier experimenters, this cotton, I repeat, was not the common cotton wool of commerce. It has been previously treated with alkali so as to remove all fatty matters, and then repeatedly exhausted with water and dilute acid, until its reaction was neutral, all soluble matters removed, and a white fibrous material as near pure cotton fiber as possible, had been obtained.

Still, in performing a crucial experiment, it was certainly important to eliminate every surface of contact except those of glass and water. Even mercury or any other possibly oxidizable metallic surface is objectionable, on account of a possible deozonation of the very minute amounts of ozonized air, which are formed by ozonation with phosphorus.

But before stating the details of this experiment, it will be well to narrate that performed by Baumann (*Zeitsch. f. physiologische Chem.* V, 250), who subsequently to my own labors effected the conversion of carbon monoxide to dioxide at ordinary temperatures by means of active oxygen.

He writes :—“A slow stream of air free from carbonic acid was conducted through a flask containing moist phosphorus ; thence into a second flask containing a mixture of 3 vols. oxygen and 1 vol. carbonic oxide, and finally through baryta water. The last remained perfectly clear after the gas had been passed through it for six hours, and after every trace of carbonic acid had been previously removed from the apparatus. On the other hand, when the mixture of carbonic oxide and oxygen was conducted into the flask containing phosphorus, and in which there must have been, according to our proposition, active oxygen, an altogether different result followed :—the baryta water presently became turbid and in an hour threw down an abundant precipitate of barium carbonate.”

Commenting upon the above, our critics say :—“Nothing is said



about stoppers or rubber connections, and it is safe to assume that, in the apparatus used by Baumann, ozone came in contact at some points with organic matter. Hence the formation of carbon dioxide is easily accounted for." \* \* \* \* "Whatever the sources of error in Baumann's experiment may have been, we are confident that a repetition with the precautions taken by us will show him that his conclusion is wrong."

It is not our duty, but that of the writer impugned, to inform us whether this confidence in his having neglected to take the most obvious and indispensable precautions in the conduct of his experiments, is well founded.

My own repetition of the original investigation was performed as follows :—

. Seven six-inch sticks of clean phosphorus were placed in a ten-liter flat-bottomed bottle, with an accurately fitting glass stopper, the bottle filled with air, and carbonic-acid-free, distilled water, and inverted in a small pneumatic trough, likewise filled with distilled water. Carbon monoxide, which had been made several days previously, and allowed to stand over distilled water in a glass gas-holder, was purified by passing through a long train of purifiers similar to those employed in connection with carbon determinations in steel, and to which additional wash-bottles containing caustic potash and baryta water had been added. Whatever carbon dioxide was present originally, was so thoroughly removed that the baryta water in the last wash-bottle remained absolutely limpid on passing sufficient carbonic oxide into the ozonator bottle to fill it nearly one-half. An equal quantity of air, after passage through the same train of purifiers, and equally well freed from the dioxide, was then introduced into the ozonator. While the bottle was still inverted in the trough, the tight fitting stopper was introduced, and it was then reverted, sufficient water being allowed to remain in the jar half to cover the phosphorus. The bottom of the bottle being flat, on distributing the sticks by gentle shaking, they were partly and uniformly submerged and exposed the largest possible amount of deoxidizing surface.

The ozonator still standing in the water of the trough, was then brought to a temperature of 24° C. by heating the trough, and kept at this temperature for six days. The antozone cloud (i. e. suspended hydrogen peroxide) produced the first day gradually subsided and the atmosphere became perfectly clear. The very gradual disappearance of these clouds, in part due to the gradual inter-

action of ozone and hydrogen peroxide with the formation of water and ordinary oxygen, and in part due to the mechanical difficulty with which the peroxide when once brought into a condition of aeriform and probably vesicular suspension goes into solution in water, strongly countenances the supposition that they are mainly and probably entirely, suspended hydrogen peroxide. The energy with which the oxides of phosphorus unite with and are absorbed by water, makes it an untenable supposition that they could, even if formed, remain for any length of time suspended in the atmosphere, so that whilst in my earlier experiments it seemed easier to suppose the clouds to be oxides of phosphorus than a non-volatile body like hydrogen peroxide in a state of aeriform suspension, yet later on the latter explanation was proved experimentally to be the correct one.

At the expiration of six days, the atmosphere contained in the bottle was withdrawn by the following manipulation, all contact with organic surfaces, except for a few seconds when the atmosphere remaining in the bottle came in contact with paraffine, being impossible.

The glass stopper was removed, a cork saturated with paraffine through which the necessary connecting tubes passed, inserted, and instantly the mouth of the bottle plunged beneath the surface of a mercury trough. This inversion carried the phosphorus and water down, so that the water covered the paraffined cork. Then the cork was quickly loosened and the aspirator being connected with the series of connecting tubes, but no air being allowed to enter, mercury was drawn up into the flask sufficient to cover the paraffined surface. The cork being forced back again, remained covered with mercury, during the remainder of the experiment.

The glass tubes were so bent, that no breaks or connections in them occurred between the atmosphere contained in the baryta water of the last wash-bottle through which the air was aspirated to take the place of the atmosphere contained in the ozonator, and the baryta water contained in the wash-bottle, through which the mixture of carbon oxides and air remaining at the close of the experiment, was finally passed. No break, except in the ozonator itself. And here it occurred above the surface of the water, the displacing air entering near the bottom, the gaseous products passing out from the upper portion of the jar. To prevent the diffusion backwards into the last wash-bottle of the entering current, of any of the atmosphere in the ozonator, a bulb was blown on one of the



lower curves of the entrance tube so as to form a mercury valve. Such a precaution was not necessary on the escaping tube, its end dipping directly beneath the surface of baryta water. This baryta water was again protected from the action of the atmosphere, by being connected with another wash-bottle containing neutral solution of potassium iodide. On now aspirating a current of air freed from carbonic acid through the apparatus, an abundant white precipitate formed in the collecting flask, and eventually a white crust formed on the sides of the entering tube beneath the surface of baryta water. When the atmosphere of the ozonator had been completely changed and no further precipitation occurred, the contents of the wash-bottle were transferred to a carbonic acid apparatus and the percentage estimated in the ordinary manner. The air used in aspirating, was freed from carbonic acid likewise. The weighings were as follows :

Potash bulbs after decomposition and aspirating.....	43.8295 grms.
Potash bulbs before decomposition and aspirating.....	43.8140 grms.
	<hr/>
Increase in weight (Carbon Dioxide).....	0.0155 grms.

There is no difficulty in obtaining a qualitative test for carbon dioxide from the white crust adherent to the entrance tube, and the amount of which should properly form a part of the 15.5 mgrms. actually weighed. On cutting off this tube with its white crust, dropping it into a test tube and allowing a drop of acid to run down the crust, it was energetically decomposed with the formation of bubbles of gas, and a drop of lime water properly supported on the under side of a glass cover stopping the exit of the test tube, became turbid.

Objections to the fact of the above experiment being conclusive as to the production of 15.5 mgrms. and upwards of carbon dioxide.

1st. That there was an interval during which at the close of the experiment the atmosphere of the ozonator was in contact with the paraffine cork. Admitting that the air in this experiment was sufficiently charged with ozone to decompose paraffine, yet as a matter of fact at the close of the experiment no ozone was present. The potassium iodide in the final wash-bottle, after acidulation, contained no free iodine. And as it should properly be objected that this negative result is inconclusive, on account of the combination of nitrogen and ozone in presence of alkaline liquid to a nitrate, it

should also be remembered that this reaction would not have been sufficiently energetic to detain every trace of residual ozone.

In my earlier experiment, in which 5 liters of air had been used and the amount of carbon dioxide formed was 14 mgrms., the amount of residual ozone was only 0.04 mgrm.

2nd. The water contained sufficient ozone in solution to decompose the paraffine submerged beneath in the instant of time before being covered with mercury. This is eminently improbable, since the establishment of the fact that ozone is soluble at all, was a very laborious matter, and was only successful after many long continued qualitative tests made with very highly ozonized oxygen. (J. Amer. Chem. Soc. I, 220.)

The same remark applies to the possibility of the dissolved ozone being converted into ordinary oxygen in contact with the mercury covering the paraffined cork.

3rd. It may be said that even if we grant that carbon dioxide was formed, it might have resulted from the combined action of ozone and hydrogen peroxide, both of which oxidants were certainly present. But inasmuch as neither of these bodies alone effects the oxidation of carbon monoxide, (and Prof. Remsen's own experiments are conclusive as to this point), and inasmuch as their mutual action is not of an oxidizing but of a reducing character, both bodies being reduced, the one to oxygen, the other to water, the above supposition is untenable.

I hold, therefore, that the fact of the oxidation of carbon monoxide to dioxide by air over moist phosphorus, has been established by a rigid quantitative and qualitative analysis. Moreover, that this oxidation is, as I have always held it to be, the proof of the existence of active oxygen, as a body antecedent to the formation of ozone.

The essential feature in my own interpretation of the phenomena studied, is that active oxygen is always formed when reduction of the oxygen molecule occurs at temperatures compatible with the stability of the ozone molecule. That nascent hydrogen is not essential to this reduction is shown by the reduction being effected by phosphorus, in which case the intervention of nascent hydrogen does not occur. Hydrogen and phosphorus are probably only the two most conspicuous of the bodies capable of reducing the oxygen molecule at ordinary temperatures, and whilst both these are perissads and this fact appears to be connected with the production of active oxygen, I am not able in the present state of experimental

knowledge to say that the perissad nature of the reducing element is an essential feature of the formation of active oxygen. As I have repeatedly stated, the essential part of my hypothesis is the reduction of the oxygen molecule at a temperature compatible with the stability of the ozone molecule. Even without chemical intervention, the silent electrical discharge will do this, and form primarily active oxygen and secondarily ozone. That such is the case is shown by the formation of carbonic dioxide when a mixture of carbon monoxide and oxygen are submitted to the silent discharge.

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## ABSTRACTS.

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Abstracts from the Journal of the *London Chemical Society*, by E. Waller, Ph. D.

**On the Constitution of some Bromine-derivatives of Naphthalene.** (Third notice.) R. Meldola (XLIII. p. 1.)

Description of the preparation of meta di brom naphthalene, Beta-di brom naphthalene, tri-brom naphthalene, and ortho-di brom naphthalene.

**On the Constitution of Lophine..** T. R. Japp. (XLIII. p. 9.) An argument against the view of Radziszewski, regarding the composition of the lophine molecule.

**Contributions to the Chemistry of Lignification.** C. F. F. Cross and E. J. Bevan. (XLIII. p. 18.)

The hypothesis of the authors previously adopted that *bastose* is a chemical whole in the sense of presenting a true combination rather than a mixture of cellulose with its own cellulosic constituent, has received further confirmation from their researches.

The close connection between these plant constituents and the trihydric phenols seems to be established.

A chlorinated derivative of *bastose* was obtained, which, though amorphous, was the same in composition when obtained from different sources, from jute, and from manilla. (*musa paradisiaca*.) The composition corresponded to the formula  $C_{32}H_{44}Cl_{11}O_{16}$ .

A note is appended remarking on the resemblance between the chloroderivatives from the alkaline liquors from Esparto boilers, and the halogen derivatives of the sacchulmic compounds recently described by Sestini. (Gazetta 1882, 292).

**On the Oxidation of Cellulose.** C. F. Cross and E. J. Bevan. (XLIII. p. 22.) A compound to which the name *oxycellulose* has provisionally been given, has been found to be one of the products intermediate between cellulose and oxalic acid by the reaction of nitric acid (of 60 per cent.) on cellulose. The amount obtained was about 30 per cent. of the cellulose used. Oxycellulose prepared from different sources, and by different methods of purification, had a constitution indicating the formula  $C_{12}H_{26}O_{16}$ . A nitro compound of this product was prepared, the examination of which by Eder's method (Ferrous sulphate) indicated the composition  $C_{12}H_{23}(NO_2)_3O_{16}$ .

**The Analysis of Certain Plant Fibres.** C. S. Webster. A table is given embodying the results of the examination of the fibrovascular bundles of several monocotyledonous plants and of the bast fibres of several decotyledonous plants. The reactions of these fibres with various reagents are also given. Also the ground upon which the several determinations included in the method of diagnosis employed, is based.

**On a Condensation Product of Phenanthraquinone with Ethylic Aceto-acetate.** F. R. Japp and F. W. Streatfield. (XLIII, p. 27.)

On mixing 100 grms. of phenanthraquinone with 90 grms. ethylic aceto acetate, and then adding 150 cc of dilute KHO (1:6) and warming gently with agitation, ethylic-phenanthraoxylene-aceto acetate is formed. The compound was purified by boiling with alcohol, washing with water, and finally recrystallizing two or three times from boiling benzene. It is deposited in tufts of fine white silky needles. Fuses with blackening and evolution of gas at  $184.5^\circ C$ . Soluble in alcoholic and acetic acid. By oxidation with chromic acid it yields phenanthraquinone.

Composition,  $C_{20}H_{16}O_4$ .

	Calculated	Mean of 3 Analyses
C	75.	74.93
H	5.	5.06
O	20.	

Mixed with amorphous phosphorus and fuming hydriodic acid and heated, a compound  $C_{20}H_{18}Cl_4$  was obtained.

Dissolved in  $KHO$ , a new dibasic acid,  $C_{18}H_{14}O_4$  was precipitated by addition of  $HCl$ . The silver and barium salts of this acid, were isolated and analyzed. These results appear to afford a strong argument in favor of Fittig's formula for phenanthraquinone as against that of Graebe.

### Note on the Preparation of Diphenylene-Ketone Oxide.

W. H. PERKIN. (XLIII, p. 35.) On heating salicylic acid with acetic anhydride, and then distilling off the acetic acid and excess of acetic anhydride, a mass was obtained which, by distillation, afforded an oily product, solidifying in the neck of the retort. On washing and purifying by recrystallization from alcohol, pale yellow needles (not, as had been anticipated of salicylic anhydride,) but of diphenylene-ketone oxide  $C_{12}H_8O_2$ , were obtained.

	Calculated.	Found.	
C.....	79.59	79.41	79.45
H.....	4.08	4.37	4.17

**On Certain Brominated Carbon Compounds Obtained in the Manufacture of Bromine.** S. DYSON. (XLIII, p. 36.) In a liquor obtained as a by-product at the works of the North British Chemical Company, chlorobromoform, as well as carbon tetrabromide and bromoform were found. The presence of the first has not before been noted.

**On Ethylene Chlorobromide, and some Compounds Obtained from it.** J. W. JAMES. (XLIII, p. 37.) A summary of the principal results is given as follows :

1. In preparing ethylene chlorobromide, by passing the gas into a solution of  $Cl\ Br.$ , it is necessary, in order to obtain a pure product and good percentage, that the chlorine be passed into the bromine at a temperature of about  $0^\circ$ , otherwise a substance is formed boiling some 3 to  $4^\circ$  higher than pure  $C_2H_4\ Cl.\ Br.$ , which is useless for the advantageous preparation of ethylene chlorothiocyanate.

2. If an aqueous solution of neutral sodium sulphite and ethylene chlorothiocyanate be brought together in direct sunlight, the sodium salt of a new acid, viz., ethylene-thiocyansulphuric acid,  $C_2H_4\ \begin{matrix} \diagup SO_2H \\ \diagdown SCN \end{matrix}$  appears to be produced.

3. By passing ammonia into an ethereal solution of chlorethyl

sulphonic chloride, no amide is formed; with ethyl sulphonic chloride, however, the corresponding amide is easily obtained.

4. By the action of neutral sodium, sulphite in aqueous solution upon ethylene dibromide or chlorobromide, isethionate of sodium is apparently produced, with evolution of  $\text{SO}_2$ , in addition to the well-known ethylene-disulphonate of sodium obtained by Strecker.

**On the Condensation Products of Cœnanthaldehyde. Part I. and II.** W. H. PERKIN, JR. (XLIII, p. 45.) A review of the most important researches on this subject is first given. The principal products of the action of KHO on cœnanthaldehyde are heptoic acid, the acid  $\text{C}_{14}\text{H}_{26}\text{O}_2$ , and the aldehydes  $\text{C}_{14}\text{H}_{26}\text{O}$  and  $\text{C}_{22}\text{H}_{42}\text{O}$ .

The products of the action of KHO, of nascent hydrogen, &c., upon these derivatives and upon cœnanthaldehyde are described. At the end of the paper a table of the different derivatives is given, as follows :

Formula.	Boiling Point.	Sp. Gr. at 15°.
Cœnanthaldehyde,	153–154°	0.8231
$\text{C}_{14}\text{H}_{26}\text{O}$ ,	277–279°	0.8494
$\text{C}_{13}\text{H}_{27}\text{COH}$ ,	266–268°	....
	(m. p. 29.05)	
$\text{C}_{13}\text{H}_{25}\text{CH}_2\text{OH}$ ,	280–283°	0.8520
$\text{C}_{13}\text{H}_{27}\text{CH}_2\text{OH}$ ,	270–275°	0.8368
$\text{C}_{13}\text{H}_{25}\text{CH}_2\text{OC}_2\text{H}_5\text{O}$ ,	285–290°	0.8680
$\text{C}_{13}\text{H}_{27}\text{CH}_2\text{OC}_2\text{H}_5\text{O}$ ,	275–280°	0.8559
$\text{C}_{13}\text{H}_{25}\text{COOH}$ ,	275–285°	....
	(at 250 min.)	
$\text{C}_{13}\text{H}_{27}\text{COOH}$ ,	300–310°	....
$\text{C}_{21}\text{H}_{40}\text{O}$ ,	310–315°	0.8744
	(at 300 min.)	
$\text{C}_{28}\text{H}_{50}\text{O}$ ,	330–340°	0.8831
	(at 200 min.)	
$\text{C}_{28}\text{H}_{54}\text{O}_3$ ,	330–340°	....
	(at 200 min.)	
$\text{C}_{28}\text{H}_{56}\text{O}_4$ ,	melts at 52–53°	....
$\text{C}_{21}\text{H}_{44}\text{O}_2$ ,	397–300°	....

The Sp. Gr. at 30° and at 35° are also given.

**On the Condensation Products of Isobutaldehyde, Obtained by Means of Alcoholic Potash.** W. H. PERKIN, JR. (XLIII, p. 90). The products obtained were as follows :

Formulae.	Bolling Point.	Remarks.
$C_{12}H_{22}O_4$	154-157°	Condensation products.
$C_{11}H_{20}O_4$ (?)	190-200°	
$C_{10}H_{18}O_4$	223-225°	
$C_{10}H_{18}O_4$	250-255°	
$C_{20}H_{38}O_4$	227-229°	
(100 mm. pressure).		
$C_{12}H_{22}O_4$	170-175°	Alcohol (and acetate) produced by the action of nascent hydrogen on $C_{12}H_{22}O_4$ .
$C_{12}H_{22}O_4(C_2H_5O)_2$	185-190°	
$C_{12}H_{22}O_4(C_2H_5O)_2$	240-242°	Acetates produced by the action of acetic anhydride on $C_{12}H_{22}O_4$ .
$C_{12}H_{22}O_4(C_2H_5O)_2$	248-252°	
$C_{12}H_{22}O_4$	217-223°	Produced by saponifying $C_{12}H_{22}O_4$ on $O_2(C_2H_5O)_2$ .
$C_{12}H_{22}O_4$	245-255°	Acid produced by the action of potash on isobutaldehyde.

**The Alkaloids of Nux Vomica. No. II—On Brucine. W. A. SHENSTONE.** (XLIII, p. 101.) The results show that there is reason to think that brucine is a dimethoxyl derivative of strychnine. Brucine, heated with hydrochloric acid, gave amounts of methyl chloride varying from 64.6 cc. to 90.2 cc. methyl chloride per gramme of brucine. Prolonged heating at low temperatures gave the highest results. Dimethoxy strychnine would yield theoretically 113 cc.

**The Behavior of the Nitrogen of Coal during Destructive Distillation: with some Observations on the Estimation of Nitrogen in Coal and Coke. W. FOSTER.** (XLIII, p. 105.) Experiments were made on a coal containing 1.73 per cent. of nitrogen. The most satisfactory results were obtained by combustion with copper oxide in vacuo. In this case it was found advantageous to place the tube in an outer tube of wrought iron, just sufficiently wide and long enough to encircle the whole length of the highly heated part. Wide tubes with large quantities of copper oxide were found to serve best. Of 100 parts of the nitrogen in the coal, it was found that the distribution was as follows:

Nitrogen of coal evolved as ammonia.....	14.50
“ “ “ cyanogen.....	1.56
“ “ present in the coal gas in the elementary condition.....	35.26
Nitrogen remaining behind in the coke.....	48.68

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100.

**Preliminary note on some Diazo derivatives of nitro-benzyl cyanide.** W. H. PERKIN. (XLIII, p. 111.)

By adding an aqueous solution of diazo benzene chloride to an alcoholic solution of nitro-benzyl cyanide freshly mixed with alcoholic potash, until the color became yellowish, a brown precipitate was obtained, which on filtering and purifying by re-crystallization from alcohol and acetic acid, proved to be  $C_{11}H_{10}N_4O_2$ . The alcoholic solution gives a violet color with alkalies, which disappears with acids.

It fuses at 201-202°. A second body fusing at 45°C, probably orthonitro benzyl chloride was also obtained in preparing the nitro-benzyl cyanide. The investigation is still in progress.

**Researches on the Induline Group.** Part I.—O. N. WITT and E. G. P. THOMAS. (XLIII, p. 112.)

The "indulines" are defined as those colored compounds formed by the action of amidazo compounds upon the hydrochlorides of aromatic amines, with elimination of ammonia.

In studying the action of amidazobenzene on aniline hydrochloride, the re-action has been found to afford several different coloring matters varying in quantities and properties, according to the temperatures at which the process may be conducted, and the manner of it. The following compounds were obtained:

Designation.	Formula.	Remarks.
Azophenine,	$C_{36}H_{29}N_5$ , melts at 236-237°	Identical with [Kunich's Azophenine.
Induline, 3 B,	$C_{30}H_{23}N_5HCl$ ,	Sky Blue color.
Induline, 6 B,	$C_{36}H_{27}N_5HCl$ ,	Dark-bluish purple.

The bases were also separated from these hydrochlorides, and analyzed. The researches on this subject are as yet uncompleted.

**On a New Method of Estimating the Halogens in Volatile Organic Compounds.** R. T. PLIMPTON and E. E. GRAVES. (XLIII, p. 119.)

The apparatus is so arranged that a stream of illuminating gas and air, mixed as in the Bunsen burner, passes over the volatile compound, taking up its vapors. The mixture is then burned under a trumpet tube, similar to that used in the Letheby apparatus for sulphur determinations in gas.



The products of combustion are drawn through U tubes, fitted with glass beads, moistened with caustic soda solution, and finally through a solution of silver nitrate, which acts as a guide to the completeness of absorption. Several analyses are quoted showing the accuracy attainable.

**A Modified Liebig's Condenser.** W. A. SHENSTONE. (XLIII, 123.)

The condenser is set vertically; just below the water jacket, an annular ledge of glass is placed inside the condensing tube, to catch the condensed liquid as it runs down the sides. This space is connected with a tube extending laterally to carry off the products of condensation. A reference to the accompanying cut is necessary for a complete understanding of the operation of the apparatus.

**On some Fluorine Compounds of Uranium.** A. SMITHELLS. (XLIII, p. 125.) By the action of HF on  $U_3O_8$  a green powder and a yellow solution are obtained. According to Ditte, hydrogen is given off in the process. The author was unable to obtain any. The green powder is stated by Ditte to be  $UO_2F_2$ , by Bolton to be  $UF_4$ . The examination showed Bolton to be correct. This powder, by heating affords a white crystalline sublimate—Alpha Uranium oxyfluoride.  $UO_2F_2$ .

The yellow solution was found to contain Beta Uranium oxyfluoride. On fusing  $U_3O_8$  with alkaline fluorides, compounds containing apparently alkaline uranates, but no fluorine except as impurity, were obtained.  $U_3O_8$  fused with  $KHF_2$  afforded  $UO_2F_2$ ,  $KF$ . The results all through are altogether at variance with the conclusions of Ditte.

**On the Volume Alteration Attending the Mixtures of Salt Solutions.** W. W. J. NICOL. (XLIII. 135.)

The conclusions are: 1. When two salt solutions which cannot experience double decomposition, are mixed, a change of volume takes place due to the different affinities of the salts for water.

2. That double decomposition takes place in solution, and that the volume change is an index, and even a measure of this.

**Two New Aluminous Mineral Species, Evigtokite and Liskeardite.** W. FLIGHT. (XLIII. p. 140).

The Evigtokite from the Greenland cryolite beds, was in white transparent crystals, quite soft. It contained

Al	16.23
Ca	22.39
Na	0.43
H, O	5.71

Indicating the formula,  $\text{Al}_2 \text{F}_6 \cdot 2 \text{Ca F} 2 \text{H}_2 \text{O}$ .

The Liskeardite from Cornwall, first described by Mr. R. Talling, in 1874, contained :

$\text{Fe}_2\text{O}_3$	7.640	
$\text{Al}_2\text{O}_3$	28.229	
$\text{As}_2\text{O}_5$	26.962	
$\text{S O}_2$	1.111	
$\text{CuO}$	1.027	
$\text{CaO}$	0.719	
$\text{H}_2\text{O}$ at ordinary temp.	4.351	
“ at $100^\circ$	10.962	$\left. \begin{array}{l} 6\text{H}_2\text{O} \\ 3\text{H}_2\text{O} \\ 7\text{H}_2\text{O} \end{array} \right\} 34.053$
“ at $120^\circ$	5.551	
“ at $140\text{--}190^\circ$	8.220	
“ with $\text{PbO}$	4.969	
<hr/>		
99.741		

The formula is put as  $\text{R}''' \text{AsO}_4 \cdot 8\text{H}_2\text{O}$ .

### On the Absorption of Weak Reagents by Cotton, Silk and Wool. By E. J. MILLS and J. TAKIMINE. (XLIII. p. 142.)

The absorption of  $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$  and  $\text{NaHO}$  were tried upon these different textures. The ratio of absorption for cotton and silk are:

	Cotton.		Silk.
$\text{H}_2\text{SO}_4$	1	:	2.6
$\text{H Cl}$	1	:	2.2
$\text{NaHO}$	1	:	2.3

Wool and silk resemble one another with  $\text{NaHO}$  but with the acids the wool takes up much more.

The resemblance between silk and cotton with these reagents is most marked, probably indicating a similarity in chemical composition between the two.

**On the Action of Chlorine on Certain Metals.** R. Cowper. (XLIII, p. 153.) With perfectly dry chlorine gas no action was observed on Dutch metal, Zinc, Magnesium, and Sodium.

Very slow action on Silver and Bismuth.

Rapid action on Tin, Arsenic, Antimony and Mercury.

With Potassium the action was very slow, a rich purple colored compound forming; by heating to fusion the action was accelerated, but the metal did not take fire until heated considerably above the fusing point. Very minute quantities of water determined the attack of almost all the above metals by the chlorine.

**Some Notes on Hydrated Ferric Oxide, and its Behavior with Hydrogen Sulphide.** L. T. Wright. XLIII. p. 156.

It was found impossible to obtain absolutely pure ferric hydrate by precipitating ferric chloride with ammonia, some basic chloride always remaining in the precipitate.

Freshly precipitated ferric hydrate suspended in water on being saturated with hydrogen sulphide, turned black and then dissolved in potassium cyanide, the reaction being:



After washing and drying at 100°C. it did not exhibit the same phenomena.

Ferric hydrate boiled with  $\text{H}_2\text{S}$  afforded some ferrous sulphate. Free sulphur always resulted from this reaction, when conducted in such a way as to imitate the action going on in gas purifiers. It appeared that 17 to 30 per cent. reacted thus:  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O} + 3\text{H}_2\text{S} = 2\text{FeS} + \text{S} + 4\text{H}_2\text{O}$  and the remainder thus:  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O} + 3\text{H}_2\text{S} = \text{Fe}_3\text{S}_4 + 4\text{H}_2\text{O}$ .

**Note on some Derivatives of Fluorene  $\text{C}_{13}\text{H}_{10}$ .** W. R. Hodgkinson and F. E. Matthews. (XLIII, p. 163) The compound used as a standing point was ortho fluorene,  $\text{C}_{13}\text{H}_{10}$  melting at 113°.

The main object was the preparation of fluorol  $\text{C}_{13}\text{H}_{10}\text{O}$ . The compounds made were: alpha dibromfluorene melting at 165°  $\text{C}_{13}\text{H}_8\text{Br}_2$ . Monobromfluorene  $\text{C}_{13}\text{H}_9\text{Br}$  melting at 101–102°. Dibromodephenylene ketone  $\text{C}_{13}\text{H}_6\text{Br}_2\text{O}$  melting at 198°. Bromdiphenylene ketone,  $\text{C}_{13}\text{H}_7\text{BrO}$  melting at 104°. The sulphonic acid,  $\text{C}_{13}\text{H}_9\text{SO}_3\text{H}$ . The barium salt of this acid containing  $2\text{H}_2\text{O}$ , and the cadmium salt containing  $6\text{H}_2\text{O}$  were analyzed.

On fusing the potassium sulphate with potash instead of fluorol, trihydroxyphenol  $C_{12}H_{10}O_3$  (melting at  $204-205^\circ$ ) and dihydroxyphenol  $C_{12}H_{10}O_2$  (melting at  $98^\circ$ ) were obtained, the methylene group having been split off.

Alpha dichlorofluorene  $C_{11}H_8Cl_2$  melting at  $128^\circ$  and its corresponding ketone melting at  $158^\circ$  were also obtained, as well as  $C_{11}H_8Cl$  melting at  $104^\circ$ . Alcoholic potash reacted with this last compound affording a red body of the formula  $C_{11}H_8Cl$ .

The sulphonic acid corresponding to the alpha dibromofluorene were prepared (melting at  $142^\circ$ ) and the barium salt  $(C_{11}H_7BrSO_3)_2Ba$  was analyzed.

**On alpha-ethyl-valero lactone, alpha-ethyl-beta-methyl-valero lactone, and on a Remarkable Decomposition of beta-ethyl-aceto-succinic ether.** S. Young. (XLIII, p. 172).

**On the Constitution of Molecular Compounds. The Molecular Weight of basic Ferric Sulphate.** S. U. Pickering (XLIII, p. 182). The experiments were conducted by means of ascertaining the unit of water removable. The choice was between the formula  $2Fe_2O_3SO_3$  (molecular weight 400) and  $Fe_2(SO_4)_3, 5Fe_2O_3$  (molecular weight 1200.) The results indicate that the latter formula  $Fe_2(SO_4)_3, 5Fe_2O_3, xH_2O$  is most probably correct.

**The Phenates of Amido Bases.** R. S. Dale and C. SCHORLEMMER. (Vol. XLIII, p. 185.) Red aurin was found to be nothing but aurin containing pararosanilin.

On dissolving equal molecules of aurin and rosanilin in alcohol, and evaporating rosanilin aurinate was found to remain. Anilin phenate  $C_6H_7N.C_6H_6O$  was obtained, fusing at  $29.5^\circ$  and boiling at  $184.5^\circ$  (uncorr.) This salt was found to be a powerful antiseptic. From an alkaline solution of aurin which, though originally red, had turned brown, the addition of an acid caused the separation of di-oxy-phenyl-ketone  $CO(C_6H_4OH)_2$ .

**On some Derivatives of Diphenyl Ketone Oxide.** A. G. PERKIN. (Vol. XLIII, p. 187.) A by-product in the preparation of this oxide (by distilling a mixture of salicylic acid and acetic anhydride) was found to be a compound  $C_{14}H_8O_3$ , fusing at  $192^\circ$  and distilling with partial decomposition. This compound, boiled with alcoholic potash, afforded an acid  $C_{14}H_8O_4$ . Both this acid and its silver salt were analyzed. The acid fused at  $275^\circ$ .

Diphenyl ketone oxide treated with nitric acid (Gr. 1.45) and a little sulphuric, yielded the di-nitro compound,  $C_{12}H_6(NO_2)_2O$ , crystallizing in satiny needles having electrical properties and fusing at  $262^\circ$ . Tin and hydrochloric acid afford with this compound the corresponding diamido-phenylene ketone oxide  $C_{12}H_6(NH_2)_2O$ , forming orange to ruby colored needles. Both of these last compounds affect polarized light.

Nordhausen sulphuric acid heated with the oxide, on dilution, neutralizing with  $BaCO_3$  &c., afforded the barium disulphonate  $C_{12}H_6O_2Ba(SO_3)_2$ , moderately soluble in boiling water, and quite stable even at  $200^\circ$ .

Bromine with the oxide afforded  $C_{12}H_6O_2Br_2$ , forming transparent needles fusing at  $210^\circ$ , and subliming a few degrees higher.

**Chemico-Microscopical Researches on the Cell-contents of Certain Plants.** A. B. GRIFFITHS. (Vol. XLIII., p. 195.) Savoy cabbages grown in a soil to which no iron compounds were added, were less healthy than those grown in a soil to which ferrous sulphate had been added. The ashes of the plants also showed more iron where it had been added to the soil, and in all cases crystals of ferrous sulphate were found by the microscope in the cells of the plants.

**On Condensations of Compounds which Contain the Dicarbonyl Group with Aldehydes and Ammonia.** F. R. JAPP. (Vol. XLIII., p. 197.) Controversial.

**On some Condensation-Products of Aldehydes with Aceto-Acetic Ether and Substituted Aceto-Acetic Ethers.** F. E. MATTHEWS. (Vol. XLIII., p. 200.)

The results given are those from an examination of,

1. The condensations of aceto-acetic ether with isobutyraldehyde, valeric aldehyde, chloral, furfural, and acrolein. The relations of di-substituted aceto-acetic ethers to benzoic aldehyde. 2. Benzoic aldehyde with aceto diethyl acetic ether, aceto dichloroacetic ether, and aceto benzliden acetic ether. Also, 3. Benzoic aldehyde with aceto-monoethyl acetic ether. The condensation takes place only in the methylene group, quite readily in the case of aceto-acetic ether, but with more difficulty with the mono and di-substitution products of that ether.

**Contribution to the Chemistry of "Fairy Rings."** J. B. LAWES, J. H. GILBERT and R. WARRINGTON. (Vol. XLIII., p. 208.)

The "fairy rings" are rings of grass, growing more luxuriantly than that in the immediate neighborhood, the ground within the ring sometimes appearing almost barren. A growth of fungi is usually observable around the outside of these rings, which increase perceptibly in size from year to year.

The theory has been advanced that the fungi assimilate nitrogen from the air, and by their death and decay, render this nitrogen assimilable by the grass. The examination of the soil within, on, and outside of the ring, shows that, though poor in nitrogen within the ring, the soil is somewhat richer in nitrogen outside of it than on the ring. The conclusion is, that the fungi have the power of assimilating the organic nitrogen of the soil to a greater extent than the grass, and that the atmospheric nitrogen has no essential connection with the phenomenon.

The report of the Anniversary Meeting of the Chemical Society, held March 30th, 1883, is here inserted p. 224.

# PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

*Regular Meeting, June 1, 1883.*

President JAMES C. BOOTH occupied the chair.

The minutes of the May meeting were read and approved.

The following gentlemen were then unanimously elected regular members of the Society :

ADOLPH HOEGLER.

Prof. P. W. BEDFORD.

WILLIS G. TUCKER, M. D.

LEWIS GORDON PAUL, D. SC.

THOS. RADCLIFFE.

The following gentlemen were then proposed for membership :

Philip S. Barker, by E. Waller, C. F. Chandler, T. S. Gladding.

C. F. McKenna, by E. Waller, A. H. Elliot, L. Pitkin.

The following papers were then read:

I. On the Nascency of Oxygen, by Dr. A. R. Leeds.

2. On Determining the Rotation of Left-handed Solutions with the German (Scheibler-Vetzke Soleil) Instrument, by George S. Eyster, Ph. D.

3. Some Notes on the Ammonia Process for Water Analysis (illustrated by the pump-well waters of Brooklyn and New York Cities), by Nelson H. Darton.

Remarks on the first paper were made by Prof. Benneman.

The third paper drew forth from Dr. Booth an earnest expression as to the importance of water analysis.

A discussion as to the influence of the depths of Brooklyn wells on their character, then followed, and was participated in by Messrs. Booth, Bartley, of the Health Board of Brooklyn; Pitkin, Alsberg, and others. The methods of analysis were also discussed by Messrs. Darton, Doremus, Waldstein, and others.

The Society unanimously ratified the choice of Martin E. Waldstein, by the Committee on Publications, to supply the place of L. H. Friedburg,

Some excellent micro-photographs of elephants' milk and cream were exhibited before the Society by Dr. Stratford.

Dr. Booth was requested, by vote of the Society, to correspond with the committee of the German Chemical Society, to see what steps might be taken in regard to the Wöhler testimonial.

The Society then adjourned.

THOS. S. GLADDING,  
*Recording Secretary.*

## ON DETERMINING THE ROTATION OF LEFT-HANDED SOLUTIONS WITH THE GERMAN (*Scheibler-Ventzke-Soleil*) INSTRUMENT.

BY GEO. S. EYSTER, PH. D.

The following plan for determining negative rotation, in the absence of an instrument graduated for left-handed solutions, occurred to me some time ago. Though seeming quite obvious, I have not seen it in print.

If we use a + quartz plate reading  $n$  degrees to the right (either used instead of one of the cover-glasses, or sunk in an outside recess of one of the brass caps, and secured in place by a thin metal ring and three screws) in connection with a — solution of less rotating power the reading will be  $N$  or  $n-x = N = -x = (N-n)$  where  $-x$  = the rotating power of the solution in degrees of the instrument. For instance :

The reading of the quartz plate is  $90^\circ$ .

The reading of the plate and solution is  $66^\circ$ , then  $-x = 66^\circ - 90 = -24^\circ$ .

Or in other words, the solution has lowered the reading from  $90^\circ$  to  $66^\circ$ ; hence is left-handed and is equal in value to the difference between  $90^\circ$  and  $66^\circ$ .

It is best to use a plate reading from  $80^\circ$  to  $100^\circ$  rather than one of much less thickness, as apart from the facility it affords for using a double normal solution for inversion, the value and equality of that portion of the scale is better under control.

I have not had a good opportunity to test the method since it occurred to me, but its success, as one acquainted with the principle of the quartz compensator will readily see, is merely a matter of detail.

## NOTES ON THE AMMONIA PROCESS FOR WATER ANALYSIS (ILLUSTRATED BY THE PUMP-WELL WATERS OF BROOKLYN AND NEW YORK CITIES.)

BY NELSON H. DARTON.

It is with hesitation that I add to the now almost too voluminous literature of water analysis. The investigations of which I wish to give results have revealed however considerable matter of interest, especially in regard to the volatile nitrogenous matters which for some time have been known to be a con-



stituent of polluted potable waters. Remsen, in his report on Boston water, in 1881, called attention to them more definitely, and Marsh quite recently has shown their influence on the results obtained in the analysis of even slightly contaminated waters.

The importance of recognition of these volatile constituents which are generally lost sight of in the ordinary method of analysis, as they pass over with the free ammonia but do not affect the Nessler reagent is, however, as yet undetermined, and by investigations of its physiological action in the concentrated state upon rabbits I have been enabled to draw conclusions which I trust will not be without value in judging the character of a potable water, as the sequel will show.

It was also very desirable to investigate its association with the other ingredients generally present in contaminated waters ; to what extent these relations change by allowing the waters to stand, and of the microscopic growths present in the waters examined ; consequently the research was extended to include all these data as completely as the facilities in my laboratory would allow, and are given in the tables.

The waters selected for the investigations were known to be polluted with sewage and decomposing vegetable matter, and highly charged with these volatile nitrogenous matters of which I wished more particularly to investigate the characteristics. These were the well waters of Brooklyn, more familiarly known as "pump waters." There are about three hundred of these wells and their pumps, most of them less than a hundred feet in depth, that yield a plentiful supply of cool, clear water even in the dry seasons. The sources of supply of these wells, or in other terms their water-sheds, are almost exclusively from water infiltrating through the cobble-paved streets and backyards of houses, then through gravel and sand, finally collecting in impervious depressions, generally of clay, underlying the city. The area drained to each pump is quite extensive, as Brooklyn is built upon the slopes of numerous hills. So far as I can find, there was no system followed in locating the pumps ; they are more abundant in the lower and older wards of the city, now populated by the poorer classes, and having with few exceptions the highest death rates of any portion.

The sources of pollution of these waters are numerous. The principal one is by infiltration—first through the filthy streets, covered with animal excreta and decomposing vegetable matter, then through the soil and subsoil, saturated with accumulated decayed and slowly

putrefying matter, whose only source of elimination is by these waters—much of this finds its way to the sea, but no small proportion of it is raised in the pumps. Another source of pollution which I am led to believe is a very considerable one, is from the loosely built sewers and carelessly-joined drainage pipes from the houses. Then, again, from the loosely-floored stables, manure pits, corner cesspools and closets in many of the backyards a great amount of sewage drains into the ground and adds to the pollution. There is naturally little or no opportunity for purification of these waters by aëration.

Three of the few remaining pumps in New York city were included in the investigation for comparison. These wells are similarly polluted to those of Brooklyn.

Thirteen wells were indiscriminately selected, numbered in the table from Nos. 75 to 88, and including the three New York City wells.

The analyses were made at three intervals, first on the same day that they were drawn; second, after standing forty-eight hours, and third, after six days. During these intervals they were kept in bottles but half-filled and loosely stoppered.

The results were obtained as follows:

*Residue.*—50 cc. evaporated, dried for three hours at 100° and weighed.

*Loss on ignition.*—The above residue was ignited for twenty minutes, cooled and weighed. Carbonic acid water was not added.

*Chlorine.*—Titration on 50 cc.

*Nitrites.*—According to Preusse and Tiemann's description of the application of Griess' test (metadiamido benzol.)

In these and other colorimetric examination the colors obtained were compared with those produced in solutions of known strength of the constituent sought.

*Nitrates.*—By the zinc-copper-couple as described by Thorpe.

*Oxygen required.*—By the moist combustion method of Wauklyn half the quantities prescribed by him being taken.

*Ammonia*, free and albuminoid, by Wauklyn's method. To obtain permanganate solution free from ammonia the following process was followed: Freshly fused potassa was allowed to cool out of contact with the air in an exhausted bell glass; this and the proper amount of permanganate were dissolved in sufficient absolutely ammonia free water, readily obtained by redistilling Ridgewood water with the proper precautions. The solution thus made

was boiled in a distilling flask with a mark on its neck whereunto it held half a litre of water at  $100^{\circ}$  c.; it was connected with a condenser, and when 10 cc. of distillate was found to be free from ammonia, the flask was filled with pure boiling water and poured into thoroughly cleansed, small hot bottles of about 100 cc. capacity, for which it was used. Frequent tests of this method revealed not even a trace of ammonia, even when distilling from a half litre and collecting the first 10 cc. of distillate.

For measuring this solution a pipett of the form that I proposed before the Society some time ago\* but with a three foot length of rubber tubing and two strong pinchcocks. Upon it were two marks, one at 10 cc. capacity, the other on the stem at 1 cc.

In every instance 100 cc. of the water was taken and the distillation conducted in 250 cc. in distilling flasks of German glass.

Precautions were taken against the disturbing influence of ammonia in the air of the laboratory by placing dishes of dilute hydrochloric acid in the niche which was devoted exclusively to the water analyses. This did not, however, appear to be necessary.

In the estimation of the volatile nitrogenous matters 100 cc. of the water were diluted, and every 10cc. of distillate diluted to 20 cc. and then divided into two portions, one of which was Nesslerized directly and confirmed the free ammonia found in the first distillation, and the remaining portion placed in a 35 cc. distilling flask joined to a miniature but effective condenser, some water and 1 cc. of the permanganate mixture added and redistilled—the 1st 10 cc. of distillate always contained all the  $\text{NH}_3$  and was Nesslerized, the difference between this and the first result being equivalent to the ammonia from the volatile nitrogenous matters. In order to determine some of its characteristics, if possible, all the waters examined were concentrated from five litres to 50 cc. This we readily accomplished by distilling at first in half litres and collecting each first 100 cc. distillate, these were mixed and the litre obtained distilled in two portions, each first 100 cc. of distillate being likewise collected; to this a calculated amount of phosphoric acid was added to hold back the free ammonia, 50 cc. was then distilled off and after redistillation was found to be quite free from ammonia. After filtration through asbestos felting 1 cc. distilled with sufficient permanganate mixture and the ammonia Nesslerized, showed that about

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\* This journal, III.—12.

two-thirds of the  $\text{NH}_3$  of the volatile nitrogenous matter had been secured and all other bodies, both chemical and organized, had been left behind.

A number of fine young rabbits were selected and sufficient amounts of these concentrations were injected under their skin with the effects detailed after each table. This it was expected would confirm a suspicion that I had long entertained that these matters were deleterious to the animal economy, and is, I think, fully justified by the results obtained.

Microscopic examinations were made of the sediments from the waters which were generally very slight; and the various forms of life found, so far as I could identify them, are detailed after the tables. In order to separate and examine the Bacilli, Micrococci and similar organisms, two methods of separation or rather concentration were tried, the first by adding osmic acid and allowing that which had been killed by its action to deposit; and the other by developing in a film of gelatine; both of these methods were found to yield quite satisfactory results, but preference was given to the latter on account of its larger yield. Before covering with the film a small amount of Cohn's developing liquor was added to the water. The dissolved gases were determined by the usual method of distillation and absorption by potash and pyrogallate of potassa. It may be well here to recall the significance of the relations of the proportions of the gases dissolved in water and their change upon standing. Water, under the most favorable conditions of purity and at  $15^\circ \text{C}$ ., will dissolve 6.3 cc. of oxygen and 11.7 cc. of nitrogen, and the relation is only disturbed to any great extent by putrefaction of matter held in solution and also by the action of ferments.

At about the time that the research was commenced Dr. A. R. Leeds in a preliminary, and as yet unpublished communication to the N. Y. Academy of Sciences, called attention to a new actinic method for determining putrescible organic matter in potable waters, which, in a number of trials he had found to yield extremely satisfactory results. Consequently it was thought desirable to include an investigation of the value of this new method.

The details of the process were orally communicated to me, and were quite closely followed. One hundred cc. of the water is mixed with the same amount of decimonal silver solution preferable the nitrate; the mixture in a closed bottle is exposed to light as long as it is acted upon, and the decomposition has been completed, the putrescible organic matter having been oxidized at the expense

of the silver, which precipitates as metal. All is then poured through a Gooch filter, and this and the bottle washed with ammonia to dissolve haloid salts. The silver adhering to the inside of the bottle and that on the filter is then dissolved in nitric acid and determined as may be convenient; its weight in millegrammes multiplied by 10 equal parts per million, and by the ratio of 108:16 equal oxygen required.

This process has so far yielded the most satisfactory of results, and promises to be a convenient, applicable and accurate method for the analysis of potable waters, if not alone, at least with the ammonia process, and when appreciable amounts of sulphides are absent.

The location of the wells and results of the physical and physiological examinations of the waters were as follows: The effects on rabbits here spoken of are the effects produced by the subcutaneous injection of a small portion of the concentrated nitrogenous matter obtained as described.

No. 75. Corner of Johnson and Lawrence streets.

Appearance in 2 ft. tube, bright and clear. Sediment slight. Microscopical examination of sediment: Sand, fibrous vegetable matter, a cypris, diatoms, and a few yellow algae.

The concentrated volatile nitrogenous matter injected under the skin of a rabbit caused violent diarrhœa, with vomiting and death in about an hour.

No. 76. Near corner Myrtle avenue and Lawrence street.

Bright and clear. Sediment considerable, containing much sand, also, *confervae*, *desmids*, *oscillatorae*, *palmaceae*, and *volvox*, two varieties of *amoeba* a *chilodon*, an *auginuillata fluvialis* and numerous diatoms.

The effect of the concentrated nitrogenous matter on a rabbit was essentially the same as with No. 75, but more intense.

No. 77. Corner of Bridge and Tillary streets.

Bright and clear. Sediment small, containing sand, fibrous vegetable matter, some diatoms and a few dead *chaetonotii*. Injection of the nitrogenous matter under the skin of a rabbit produced a slight transitory diarrhœa.

No. 78. Corner Sumner avenue and Bainbridge street.

Strong odor of brewery swill, develops that of  $H_2S$  on standing. Sediment abundant, dark colored, containing numerous algae and animalculæ. The effect on a rabbit was to produce diarrhœa,

cramps, vomiting and death. A similar effect, though not so rapidly fatal, was produced on a cat.

No. 79. Corner Johnson and Bridge streets.

Water clear and bright. Sediment small and containing scarcely anything worthy of mention. Effect on a rabbit similar to that of the previous one.

No. 80. Corner Chapel Place and Bridge street.

140 feet deep. No sediment. Effect on a rabbit slight transitory diarrhœa.

No. 81. Near corner of Irving Place and Fulton avenue.

Clear and bright. Sediment slight, consisting of sand, *diatoms*, *algae*, several *cypri*, *chilodons* and *amoebæ*. Effect on a rabbit, violent diarrhœa followed by slow recovery.

No. 82. Corner Bridge and Nassau streets.

Water clear. Sediment large, containing sand, algae and monads. The bacteria obtained by filming when injected under the skin of a rabbit caused diarrhœa and death. The same effect was produced by the concentrated nitrogenous matter.

No. 83. Corner Fulton avenue and Carleton.

Water clear. Sediment inconsiderable. In this water the proportion of total ammonia was much less than the sum of the five and albuminoid together. It probably contained some substances oxidized by the permanganate to nitrates. The tests were duplicated with the same results.

No. 84. Corner Duffield and Johnson streets.

Water clear. Sediment moderate, containing sand, numerous *diatoms*, *chilodons*, etc. Effect on a rabbit, violent diarrhœa, etc. Death in two days.

Some of this concentrated nitrogenous matter was also injected under my own skin. The effects were similar. Diarrhœa was produced in two hours, and continued for two days.

No. 85. Hanover Place near Fulton street.

Water clear. No perceptible sediment. Effect on a rabbit, intermittent diarrhœa lasting for about a week. The effect on my assistant was to produce a tendency to diarrhœa lasting four days.

No. 86. New York City, corner Thames and Church streets.

Water clear. Slight odor of acetylene, which soon disappeared. Contained no volatile nitrogenous matter. Effects on a rabbit nil.

No. 87. New York City, corner Dey and Greenwich streets.

Water clear. Much sediment. Both the sediment and the volatile nitrogenous matter produced violent diarrhœa, and vomiting,

with rabbits, which in the case of the sediment terminated in death.

No. 88. Corner Church and Cedar streets.

Similar to the last. Sediment contained vorticella and microstoma. The volatile nitrogenous matter injected under my own skin produced diarrhœa and vomiting from which I took a week to recover.

To determine whether the above results were exclusively due to the volatile nitrogenous matters and whether they existed in the water as such, or were formed during distillation, further experiments were tried from time to time during the research. That these volatile nitrogenous matters preëxisted in the waters was, I thought, very probable. To ascertain this the waters were, after being filtered through asbestos plugs (in all other respects they were as when drawn from the wells), injected in considerable quantity under the skins of rabbits. Symptoms were produced very similar to those induced by the concentrated waters, and the distillates from the concentrated waters which had been found not to contain or develop the volatile nitrogenous matters. Well No. 86 greatly polluted, otherwise produced not the slightest discernable effect upon the animal's health, and when the free ammonia was allowed to accumulate in another portion and concentrated till it became two parts per million, on injection it produced exactly similar results.

In order to determine the effect of the less volatile portion of a water, some of that which had been filtered through the asbestos plugs of our other experiment, was distilled at a very low pressure at a temperature of about 84° C., as previous experiments had shown that all the readily volatile matters of this water were easily separated from it by heat, applied if for even a short time. That which remained and was injected in the rabbit produced little or no discernable effect. While in this and the first injectings, bacterial matter might have been implicated, the latter experiment would have failed on account of their sterilization by the heat applied. Yet the volatile matters, in which in every instance I am sure there were no bacteria present, produced more or less violent diarrhœas with the exception, I might again mention, of that from well No. 77, which, although when concentrated, contained 7 parts per million, an amount equal to that in many of the others, produced no diarrhœa in the rabbit into which it had been injected. The only manner by which I can account for this is by assuming that the



term "volatile nitrogenous matters" is a very elastic one, and includes both virulent and harmless amine compounds. Of the former, those of phenol, toluol, etc., and of the latter, ethyl, etc. This is a part of the subject, however, that I trust to be enabled to further elucidate, while it suffices in the present communication to show that the volatile nitrogenous matters accompanying the other pollutants in a sewage contaminated water probably are injurious to health, and that in an examination of a water to determine its potability, their presence and amount should not be lost sight of.

N. Y. LABORATORY, 133 WATER ST.

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## ABSTRACTS.

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Abstracts from the *Journal of the London Chemical Society*: E. Waller, Ph.D.

**On the Estimation of Hydrogen Sulphide and Carbonic Anhydride in Coal Gas.** By Lewis T. Wright. (Vol. XLIII. p. 267.)

Experiments with an absorption apparatus of the Frankland-Ward type showed that a manganese peroxide bullet introduced some gases with it mechanically, unless previously submitted to the action of a stream of coal gas for some hours, after which it removed a small amount of gas mechanically. Copper sulphate dried at 150° absorbed some gas mechanically. Copper phosphate moistened with syrupy phosphoric acid gave tolerably good results.

In experiments with absorption tubes, the amount absorbed being weighed, cupric sulphate was found to be objectionable for the following reasons: The sulphuric acid set free reacts with the hydrocarbons in the gas,—sulphurous acid will always form (whether the sulphate has been dried as recommended by Fresenius for four hours at 150°-160° or not.) The reagent also is not very sensitive to small amounts of H<sub>2</sub>S. Ferric and manganic oxides are not sufficiently sensitive. Mercuric oxide steadily loses weight in a current of pure coal gas. Cupric phosphate was the most satisfactory. It was prepared by dissolving 2 lbs. Na<sub>2</sub>HPO<sub>4</sub> (crystals?) in 1 gal-



lon of water and  $2\frac{1}{2}$  lbs.  $\text{CuSO}_4 \cdot 7 \text{H}_2\text{O}$  in  $1\frac{1}{2}$  gallons of water and mixing the solutions with vigorous stirring.

The precipitate was washed by decantation and then dried at  $100^\circ$ . Before using, this reagent requires to be completely "saturated" with pure coal gas, by slowly passing through the U tube containing it about 3 feet of gas free from  $\text{H}_2\text{S}$ .

The U tubes used were glass stoppered, and connected by tubes carrying stop cocks. The train used consisted of the meter next a 12 inch U tube containing pumice soaked in syrupy phosphoric acid—to remove ammonia next a tube filled with (neutral) calcium chloride, then a 6 inch tube, one leg of which was filled with the cupric phosphate, the other with neutral calcium chloride, and next a 6 inch U tube having one leg filled with moist soda lime, the other with calcium chloride. The results were slightly higher than when the same gas was caused to yield the  $\text{H}_2\text{S}$  &  $\text{CO}_2$  together to soda lime.

**Contributions to the Chemistry of the Cerite Metals.** B. Brauner. (Vol. XLIII. p. 278).

Didymium oxide carefully purified, was converted into sulphate. The mean of five separate determinations gave an atomic weight of 145.42 for Di.

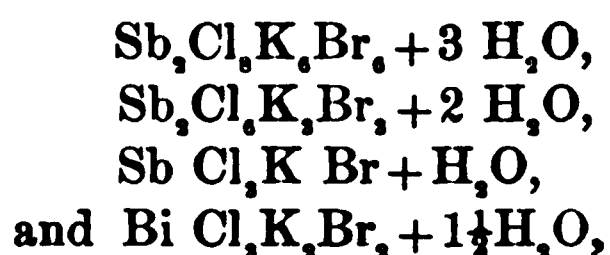
Another lot of didymium oxide when precipitated fractionally gave atomic weights ranging from 144.3 to 149.4, the last fractions giving the higher figures, indicating that previous determinations of the atomic weight 147.2 (Cleve) and 146.58 (Brauner) were probably high in consequence of the presence of at least one other element called provisionally (Di. gamma).

An examination of the absorption bands of a solution of the oxide having the highest atomic weight indicated the probability of the identity of this with samarium which probably has an atomic weight of 150.

Oxides of the yttrium metals, yttria, holmia, thulia, erbia and probably terbia, were found to exist in cerite.

**Some Compounds of Antimony and Bismuth Containing two Halogens.** R. W. Atkinson. (Vol. XLIII. p. 289).

The preparation of



are described, and analytical results given.

The first mentioned salt when heated to between 200 and 300°, loses antimonious fumes, the antimony carrying with it half the bromine and half the chlorine, which procludes the theory that the constitution of the salt is  $2 \text{ Sb. Cl}_3, 6 \text{ K Br. 3 H}_2\text{O}$ .

**Crystallographic Examination of the Crystals of Antimonio Potassic Chloro Bromide.** R. H. Solly. (Vol. XLIII. p. 293.) Examination of the crystals of the salt first described in the preceding paper.

**On the Gases Evolved During the Conversion of Grass into Hay.** P. F. Frankland and F. Jordan. (Vol. XLIII. p. 294.)

The conclusions reached are :

1. That comparatively dry grass soon evolves considerable quantities of gas consisting almost wholly of carbonic anhydride, and accompanied by mere traces of hydrogen and hydrocarbons.

2. That the evolution of gas takes place with almost equal rapidity in atmospheres composed of air, carbonic anhydride, oxygen or hydrogen ; and that excepting when the atmosphere contains oxygen, in which case a notable proportion of nitrogen accompanies the carbonic anhydride, the composition of the gas evolved is much the same.

3. That when the decomposition of grass takes place under water, large volumes of gas are evolved which are characterized by the presence of a notable proportion of hydrogen. This hydrogen is doubtless due to the lactic fermentation induced by bacteria ; acetic, lactic, and probably propionic acids, together with bacteria, being found in the water in which the grass was immersed.

**Note on an Apparatus for Fractional Distillation under Reduced Pressures.** L. T. Thorne. (Vol. XLIII. p. 301.) Requires the accompanying illustration to be intelligible.

**Notes on the Condition in which Carbon exists in Steel.** Sir F. Abel and W. H. Deering. (Vol. XLIII. p. 303.)

First series of experiments. From one lot of steel (cold rolled) discs were taken, as received, after annealing, and after hardening. The process pursued in hardening and annealing are described. Treatment with a cold saturated solution of potassic dichromate to which  $\frac{1}{2}\%$  of its volume of pure conc.  $\text{H}_2\text{SO}_4$  had been added, left undissolved, dark particles appearing sparingly under the microscope, attracted by the magnet, containing in the case of the cold rolled, and annealed discs, iron and carbon in about the proportion of

$\text{Fe}_3\text{C}_2$ , nearly all the carbon remaining in the residue. With the hardened disc only about one-sixth of the carbon remained in the residue, and the proportion of iron to carbon was much less with the others. The use of a larger proportion of  $\text{H}_2\text{SO}_4$  in the bichromate solution seemed to break down the carbide, and dissolve the greater part of the iron.

Second series. Steel which had been melted from cemented blister steel and subsequently cold rolled and cross cold rolled, with annealing between the various rollings, was tested in a similar manner. The solutions used were :

Preparation 1. 0.8, the strength of Preparation 2.

2. Cold saturated solution of  $\text{K}_2\text{Cr}_2\text{O}_7$  (99 grms. per litre) containing 0.9 grms. pure conc  $\text{H}_2\text{SO}_4$  to each grm. of bichromate.

3. 0.44, the strength of Preparation 2.

4. 1.44 in one case and 1.65 in another, the strength of Preparation 2.

With the first three preparations 13.25 to 15.34 per cent. of carbide remained undissolved as a black powder attracted by the magnet, containing iron and carbon in the proportion for  $\text{Fe}_3\text{C}$ . Preparation 4. was evidently too concentrated for the carbide to resist decomposition by it.

The results (which are regarded only as preliminary) tend to confirm the view that carbon in steel exists as an iron carbide distributed through the mass.

### **On the Spectrum of Beryllium with Observations Relative to the Position of that Metal Among the Elements.**

W. N. Hartley. (Vol. XLIII. p. 316.) A map of the spectrum is given. The conclusion is that "the spectrum of beryllium exhibits no marked analogy with the calcium, the magnesium or the aluminium spectra, all of which are members of well defined homologous series. There is nothing similar to the boron, the silicon or carbon spectra, nor to those of scandium, yttrium or cerium. The spectrum of lithium is the one most allied to that of beryllium in the number, relative position and intensity of lines. \* \* \* I am therefore led to the conclusion that beryllium is the first member of a dyad series of elements, of which in all probability calcium, strontium and barium are homologues."

**On a New Oxide of Tellurium.** E. Divers and M. Shimosé. (Vol. XLIII. p. 319.) By heating the compound of sulphur trioxide and tellurium in vacuo, until decomposition ensues,  $\text{SO}_2$  and a

new oxide of tellurium were obtained. As thus prepared it resembles charred cork, but must be purified, by washing with dilute sodium carbonate, from sulphur oxides and tellurous acid. The oxide seems to be obtained in small quantities by the action of water on the sulphoxide, and also by pouring gradually the well-known purple red solution of tellurium in sulphuric acid into a large volume of water.

Its composition may be expressed by the name and formula *Tellurium monoxide*  $\text{Te O}$ , but its constitution is probably more complex than is thereby indicated. To prove the individuality of the compound, the action of reagents on, and the general properties of a mixture of pure tellurium with tellurium dioxide, were contrasted with those of the new oxide. The physical characters and action with reagents were altogether dissimilar.

**On Tellurium Sulphoxide.** E. Divers and M. Shimosé. (Vol. XLIII p. 323.)

The compound was discovered independently of R. Weber (Jour. Pr. Chem. XXIV. 218), the printed copy of his results having reached the author before their own research was completed. The properties of the substance were found to be as described by Weber with the exception that it was not found to be unstable as stated by him. The analyses indicated  $\text{SO}_2\text{Te}$ ; the formula also assigned by Weber, but the most of the results gave an excess of  $\text{SO}_2$ , probably present as an impurity. By prolonged heating in vacuo at  $35^\circ$  or almost instantaneously by heating to  $90^\circ$  the color changes from red to a bright fawn brown. This form has been called by the authors *beta* tellurium sulphoxide, as no change of composition occurs. Heated still further, to  $130^\circ$  the compound softens and shrinks, at  $180^\circ$  it begins to decompose  $\text{SO}_2$  being evolved, the  $\text{TeO}$  described in the previous paper remaining. The decomposition is nearly complete at  $230^\circ$ .

**On a New Reaction of the Tellurium Compounds.** E. Divers and M. Shimosé. (Vol. XLIII. p. 329.) Sulphuric acid can hold in solution but small amounts of  $\text{TeO}_2$  or its sulphate. But if this is poured upon zinc and the escaping hydrogen, containing hydrogen telluride, is passed into a solution containing tellurated sulphuric acid red tellurium sulphoxide makes its appearance in the liquid. By prolonged action of the current of gas, a brown compound, probably hydrogen pertelluride, precipitates, with forma-

tion of  $\text{SO}_2$ , which can be detected by passing the gas through potassium permanganate.

Abstracts from the Comptes Rendus, by A. Bourgougnon.

**Action of Anhydrous Aluminium Chloride upon Acetone.**

By E. Louise. (No. 14, Oct. 2, 1882.)

Amongst the most volatile products found during the reaction, there is mesityl oxide, boiling point,  $128^\circ\text{--}130^\circ\text{C}$ ; composition  $\text{C}_6\text{H}_8\text{O}$ ; vapor density, 3.51. The less volatile product contains Phoron, boiling point,  $195^\circ\text{--}196^\circ\text{C}$ , composition  $\text{C}_6\text{H}_8\text{O}$ .

**Upon some Combinations of Disulphide and Diselenuride of Tin.** By A. Ditte. (No. 15, Oct, 9, 1882.)

By the action of soluble sulphides upon the sulphides or selenurides of tin, crystallized salts analogous to stannates are obtained, in which selenium and sulphur take the place of oxygen.

**Fermentation of Nitrates.** By M. Gayon & Dupetit. (No. 15, Oct. 9, 1882.)

**Upon the Reduction of Nitrates in the Soils.** By P. P. Dehérain and Maquenne (No. 16, Oct. 16, 1882.)

Under certain circumstances nitrates are reduced in the soils with production of nitrous oxide gas. The reduction of nitrates occurs only in soils containing a large amount of organic matter. The nitrates are not reduced when the atmosphere of the soils contains oxygen.

**On Alunite.** By P. Guyot. (No. 16, Oct. 16, 1882.)

The composition of alunite, from the mines of "La Tolfa," is not constant; the amount of alumina and potassium sulphate being variable. Crystallized alunite sometimes contains 30 per cent. of base, whilst sometimes the quantity of silica is so large that the proportion of base is reduced to 17.5 per cent. The average composition of alunite generally corresponds to—

Alumina	27.60
Sulph. acid	29.74
Potassium Oxide	7.55
Water	11.20
Iron	1.20
Silica	22.71
	<hr/>
	100.00

**Chronic Poisoning by Antimony.** By C. de Poncy and C. Livon. (No. 16, Oct. 16, 1882.)

**Separation of Gallium.** By L. de Boisboudran. (No. 17, Oct. 23, 1882.)

Methods for the separation of gallium from tin and antimony.

**Determination of the Atomic Weight of Thorium.** By L. F. Nilson. (No. 17, Oct. 23, 1882.)

The determinations have been made with the sulphate  $\text{Th}(\text{SO}_4) + 9 \text{H}_2\text{O}$ . The average of six determinations gives the number 232.43. In another series of experiments four determinations have given the number 232.3.

**Upon the Reduction of Nitrates in Soils.** By P. P. Dehérain and L. M. Maquenne. (No. 17, Oct. 23, 1882.)

Nitrates are not reduced in soils which have been heated. In the experiments the earth was placed in closed tubes and submitted to a temperature of  $110^\circ$ – $120^\circ$  C. When the soils contain vapors of chloroform, the nitrates are not reduced.

The nitrates are reduced when some fresh earth is mixed with earth which had been previously heated.

**Separation of Gallium.** By L. de Boisboudran. (No. 17, Oct. 23, 1882.)

Separation of gallium from tin and antimony.

**Chemical Studies on the Sugar-Beet.** By H. Leplay. (No. 18, Oct. 30, 1882.)

**A Rapid Method for the Determination of Salicylic Acid.** By A. Rémont. (No. 18, Oct. 30, 1882.)

A modification of the process which consists in extracting salicylic acid with ether, and comparing the coloration given with perchloride of iron with the color obtained with standardized solutions of acid and the salt of iron.

**Upon a Hydrate of Molybdic Acid.** By F. Parmentier. (No. 19, Nov. 9, 1882.)

The yellow crystals deposited on the sides of the bottles containing solutions of ammonium molybdate in nitric acid are formed by a hydrate of molybdic acid  $\text{MoO}_3 \cdot 2 \text{H}_2\text{O}$  very sparingly soluble in water and acids.

**Upon Monochlorinated Allylic Alcohol (Alpha)  $\text{CH}_2=\text{CCl}-\text{CH}_2(\text{OH})$  and its Derivatives.** By L. Henry. (No. 19, Nov. 6, 1882.)

Monochlorinated allylic alcohol (alpha)  $\text{CH}_2=\text{CCl}-\text{CH}_2(\text{OH})$  is obtained by the action of a diluted solution of potassium oxide upon epibichlorhydrin  $\text{CH}_2=\text{CCl}-\text{CH}_2\text{Cl}$ . It is a colorless liquid, sp : gr : at  $19^\circ\text{C}$  1.164 ; boiling point,  $136^\circ\text{C}$ . Soluble in water.

Derivatives.—Acetic ether  $\text{CH}_2=\text{CCl}-\text{CH}-\text{CH}_3(\text{C}_2\text{H}_5\text{O}_2)$  produced by the action of acetyl chloride, boiling point  $145^\circ\text{C}$ .

Bromhydric ether  $\text{CH}_2=\text{CCl}-\text{CH}_2\text{Br}$ , produced with phosphor-tri-bromide, Ph Br, boiling point  $120^\circ\text{C}$ .

Sulphocyanate produced by the action of the chloride (alpha)  $\text{CH}_2=\text{CCl}-\text{CH}_2\text{I}$  upon potassium sulphocyanate, boiling point  $180^\circ\text{C}$ . Combined with ammonia it produces monochlorinated thiocinnamin, melting point  $90^\circ\text{C}$ .

**Chemical Studies of Sugar Beet.** by H. Leplay. (No. 19, Nov. 6, 1882)

**Upon the Reduction of Nitrates in the Soils,** by P. P. Dehérain and M. Maquenne (No. 19, Nov. 6, 1882.) The authors have succeeded in determining the nature of the ferment reducing the nitrates in the soils. Some earth has been mixed with a solution of sugar (1 per cent.) and some potassium nitrates.

The gases evolved were composed of

Carbonic acid	. . . . .	80. 5.
Nitrous oxide	. . . . .	8. 2.
Nitrogen	. . . . .	11. 3.

The water collected with the gases had a strong odor of butyric acid and contained a large quantity of "bacillus amylobacter" these vibrios having most probably produced the fermentation in the earth.

**Chemical Studies on Sugar Beet,** by H. Leplay. (No. 20, Nov. 13, 1882.)

**Decomposition of Phosphate at a High Temperature,** by H. Grandeau. (No. 20, Nov. 13, 1882.)

An inquiry into the conditions by which aluminium phosphate is decomposed at a high temperature by potassium sulphate.

**Upon Cœnocyamine,** by E. J. Maumené. (No. 20, Nov. 13, 1882.)

Oenocyanine is the coloring matter of black grapes and red wines, it is colorless in grapes before their maturation, it turns of a dark blue by exposure to air.

**Decomposition of Hydrogen Dioxide by Fibrin, by A. Béchamp.** (No. 20 Nov. 13, 1882.)

Pure fibrin freshly extracted from blood decomposes hydrogen dioxide, during this reaction the fibrin is modified, and after forty-eight hours of contact, it has no longer the power of decomposing hydrogen dioxide, if even put into contact with a fresh solution, it also has sustained a loss of weight.

A drop of hydrocyanic acid stops at once the decomposition of hydrogen dioxide by fibrin, and this acid is oxidized.

**Researches on Lead Iodide, by M. Berthelot.** (No. 21, Nov. 20, 1882.) Thermochemistry.

**Production of Crystallized Uranates, by A. Ditte.** (No. 21, Nov. 20, 1882.)

Sodium Uranate is obtained by the fusion in a platinum crucible of sodium chloride and green oxide of uranium, insoluble in water, easily soluble in soluble acids.

Calcium Uranate is produced by the reaction of calcium chloride upon green oxide of uranium. Strontium and barium uranates are produced by the same method. Yellow needles insoluble in water, soluble in diluted acids.

**Mannitic Anhydride, by A. Fauconnier.** (No. 21, Nov. 20, 1882.)

Is obtained by dry distillation in a vacuum of mannite, it is a colorless liquid, boiling at 160-190° C, composition  $C_6H_{10}O_4$ , very soluble in alcohol and water, insoluble in ether. Heated with acetic anhydride it gives  $C_6H_8O_4(C_2H_5O)_2$ , a viscous liquid, nearly colorless, boiling point 197-198° C.

Phosphorus oxychloride is without action upon  $C_6H_8O_4$ , but phosphorus perchloride transforms it into  $C_6H_8O_4Cl_2$ , having the form of hexagonal tables, very soluble in ether, less soluble in alcohol and benzine, insoluble in water, boiling point 143° C.

**Chemical Studies on Corn, at different Stages of its Vegetation, by H. Leplay.** (No. 23, Dec. 4, 1882.)

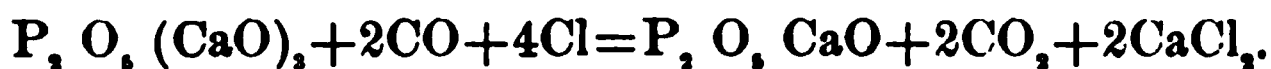
**Upon a Method to Transform Tricalcic Phosphate into Chlorinated Compounds of Phosphorous, by T. Ribau.** No. 23, Dec. 4, 1882.)



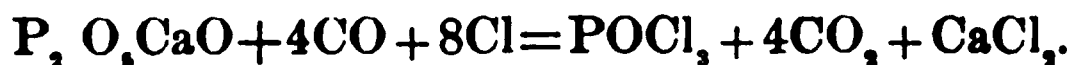
When a current of chlorine and carbon monoxide is directed upon a mixture of carbon and tricalcic phosphate, the phosphate is transformed at a low temperature into phosphorus oxichloride  $P_2O_3Cl_2$  with production of calcium chloride and carbonic dioxide.



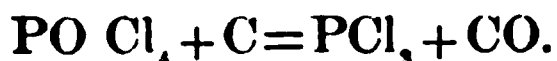
This equation represents the final result of the reaction, but in reality it can be divided into two distinct phases. First, chlorine and carbon monoxide transform the tricalcic phosphate into metaphosphate and calcium chloride without production of free phosphoric acid.



Second, the gaseous mixture changes the metaphosphate into phosphorus oxichloride.



Phosphorous oxichloride distilled over red hot charcoal is transformed into phosphorus trichloride.



#### **A New Hydrocarbon**, by E. Louise. (No. 23, Dec. 4, 1882.)

Benzylmesitylen is obtained by the reaction of benzyl chloride upon mesitylen in contact with anhydrous aluminium chloride.

**Volta Prize.** A prize of fifty thousand francs is offered and will be presented by the "Académie des Sciences," in December 1887, to the most successful discoverer of means tending to apply cheaply electricity as a source of light, heat, chemical action, mechanical power, transmission of dispatches or medical treatment. This concourse is open to the savants of all nationalities, and will close June 30, 1887.

Abstracts from *Berichte der Deutschen Chemischen Gesellschaft*, by Percy Neymann, Ph.B.

**On the Preparation of Diphenyl—keton-oxide.**—W. H. Perkin (vol. XVI. 339). In endeavoring to obtain the anhydride of salicylic acid this acid was heated with acetic acid anhydride. The acid is dissolved while acetic acid and acetic acid anhydride distil over. A thick liquid remained in the retort. When subjected to distillation a quantity of an oily substance passed over which solidi-

fied crystalline in the neck of the retort. This after being washed with alcohol and recrystallized from this gave figures from the analysis corresponding to the formula  $C_{11}H_8O_2$ . The product is evidently the same as that obtained by Merz and Weith by oxidation of methylen-diphenyl-oxide and that obtained by Richter by distillation of salicylate of potassium with phosphorus oxychloride.

**On Sulphocyanpropimin.**—J. Tscherniac and C. H. Norton (vol. XVI. p. 345). When monochlor-aceton and ammonium sulphocyanate act upon each other in alcoholic solution, the sulphocyanate of a new base is formed.



Investigation has shown that this reaction takes place in two phases ; at first sulfo-cyan-aceton is formed :



but this latter is caused to decompose by the presence of sulphocyanate of ammonia, a sulphocyanate  $\text{C}_2\text{H}_5\text{N}_2\text{S.HSCN}$  being formed while water is separated :



The authors propose the term sulphocyanpropimin for this new base  $\text{C}_2\text{H}_5\text{N}_2\text{S}$ .

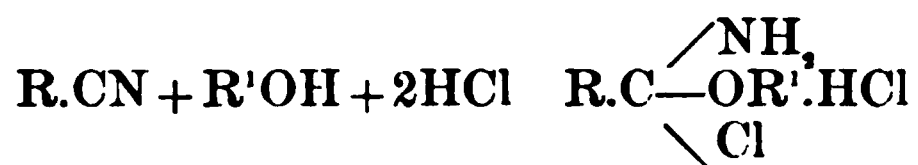
**On Sulphocyanacetone.**—J. Tscherniac and R. Hellon (vol. XVI. p. 348). 175 grms. of sulphocyanate of barium are dissolved in 525 grms. of alcohol and 100 grms. of monochloracetone are added. The mixture is left to itself several days and when no more precipitate forms this is filtered and washed with alcohol. The alcoholic solution evaporated on a water-bath leaves crude sulphocyanacetone as a syrupy mass ; this is boiled with ten times its bulk of water and the water solution is left to itself for 24 hours. The clear supernatant liquid is decanted and evaporated on a water bath. An oily mass separates. Washing and drying over sulphuric acid finally gives the pure sulphocyanacetone. Its formula is  $\text{CH}_3\text{SCN.CO.CH}_3$ . It is an oil, only slightly colored when first made, but acquires a deep red color when exposed to the air.

**On a New Industrial Method of Preparing Paper Pulp.**—(Vol. XVI., p. 350.) G. Archibald. Wood or straw is cut in pieces, macerated with milk of lime transferred, to a digester after

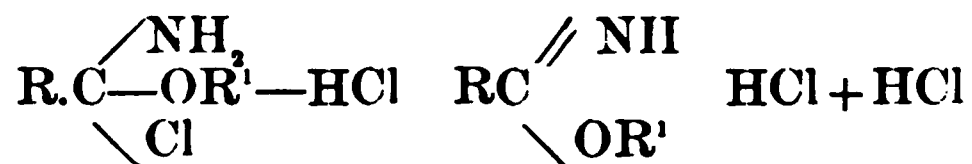
12 hours and saturated with sulphurous acid, at the same time applying a pressure of five atmospheres for one to two hours. The material is then washed with water and again treated under pressure with three per cent. calcium chloride and half per cent. aluminium sulphate. After these substances have been washed out, the pulp resembles cotton in appearance, and can be employed for manufacturing finer grades of paper at once. The process requires about three hours after the treatment with milk of lime. The process is patented in the United States and Canada.

**On the Occurrence of Methyl Alcohol in the Products Obtained from Colophonium by Dry Distillation.**—(Vol. XVI., p. 351.) Werner Kelbe and J. Livoff. Among the products obtained by dry distillation of colophonium is a small quantity of a liquid containing considerable quantities of acetic acid and in very small quantities higher homologues of this acid. Based upon the fact that methyl alcohol is produced by the dry distillation of numerous organic substances this liquid was examined for and found to contain methyl-alcohol, but not in sufficient quantity for practical purposes.

**On the Conversion of Nitrils into Imides. The Behavior of Hydrocyanic Acid and of Ethelencyanide to Hydrochloric Acid and Alcohol.**—A. Pinner. (Vol. XVI., p. 352.) As previously described, when hydrochloric acid is introduced into a mixture of equal parts of a nitril and an alcohol an amido-chlor-ether is first formed :



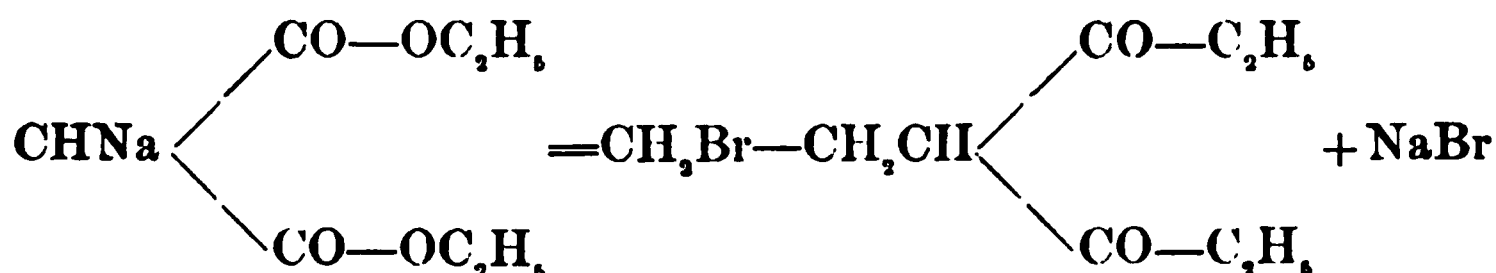
this is, however, very unstable, soon loses a molecule of hydrochloric acid and is converted into the hydrochlorate of an imido-ether :



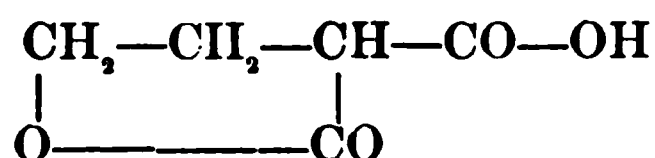
At the time these investigations were made, the nitril of formic acid, hydro-cyanic acid, was also experimented upon, but the reaction ensued in a totally unexpected manner. By introduction of hydrochloric acid gas into an absolute alcoholic solution of hydrocyanic acid, free from water, energetic detonations took place, and by par-



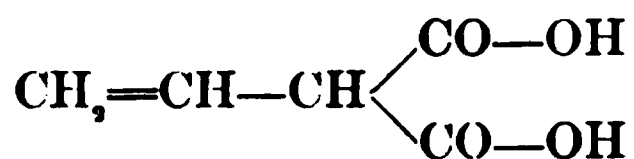
lowed to act upon sodium-malonic-acid-ether (1 Mol.). The reaction was supposed to take place :



thus forming a bromidized ethyl-malonic-acid-ether and by saponification of this the carbon acid of butyro-lactane :



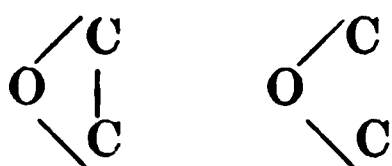
The crystalline acid obtained had the formula  $\text{C}_6\text{H}_6\text{O}_4$  but from its salts it was found to be the isomeric unsaturated acid of the above lactonic acid.



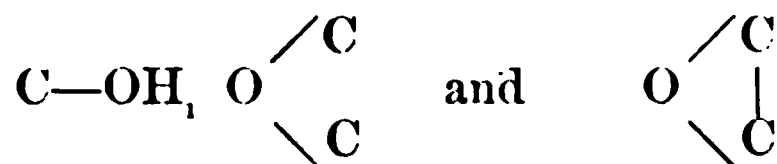
**Microscopic Examination of Printed Cotton Goods.** (Vol. XVI., p. 455). RICHARD MEYER. The author makes use of the microscope to ascertain whether the dye as such has been produced inside of the fibre or whether it has been employed already formed and fixed by means of albumen. Both methods can be employed for the same dye. For example, a method much employed is to impregnate the fibre with a soluble lead salt, to precipitate this as hydroxide, carbonate or sulphate and then to convert it into the fiery reddish-yellow, basic chromate of lead. In combination with steam dyes it may, however, be much more convenient to print the chrome-orange already precipitated with albumen and fix it by steaming. The method of detecting the difference the author gives thus : If the fabric is macerated using a needle so that the single fibres can be isolated, these will appear equally colored throughout their entire mass if they have been colored by a dissolved dye. The characteristic form of the fibre remains unaltered, and it can be plainly seen that the coloring matter is deposited equally in the interior of the fibre. When the albumen method has been used the fibre appears entirely without color. At various points dyed bits of coagulated albumen will appear gummed to the fabric, and some of these particles will be found isolated in consequence of the maceration.

**New Methods of Forming Pyro-Sulphuryl-Chloride and Chloro-Sulphonic Acid.** G. BILLITZ and K. HEUMANN. (Vol. XVI., p. 483). 140 grms. of chlorosulpho acid and 130 grms. of pentoxide of phosphorus were heated with the reversed condenser. After the evolution of hydrochloric and sulphurous acid ceased the mass was distilled at 135–150°. This sinks in water while chlorosulpho acid unites with water, accompanied by a hissing noise. 93 per cent. of the theoretical amount of pyrosulphuryl chloride were obtained. The formation of pyrosulphuryl chloride from chlorosulpho acid by means of pentoxide of phosphorus agrees with the formation of pyrosulphuric acid from sulphuric acid by means of a dehydrating agent :

**Contributions to the Knowledge of Hydroxylamin Reactions.** E. NÄGELI. (Vol. XVI., p. 494.) The author, by a series of experiments, finds it a valuable truth that hydroxylamin can be employed in general and easily whether in compounds which contain carbon, hydrogen and an atom of oxygen the group C=O or another group, such as

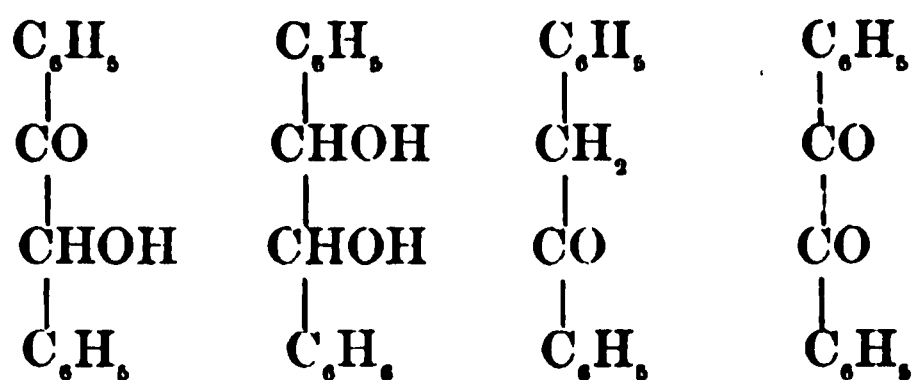


etc., is present. In order to determine by the *non-occurrence* of a reaction when hydroxylamin is employed that it contains no aldehyde- or ketone group, and on the other hand to accept the formation of an isonitroso compound as a proof of the presence of acetone or aldehydicarbonyl, it was necessary to try compounds of known constitution. This was done by the author and the law was found to be correct. All ketones so tested gave acetoximes; but the compounds with the groups :



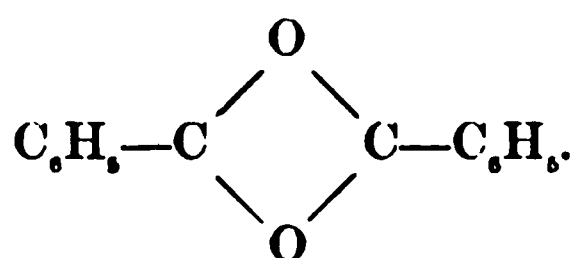
behaved indifferently to hydroxylamin such as alcohol, ether, ethylenoxyd.

**On Benzil.** MAX WITTENBERG and VICTOR MEYER. Vol. XVI., p. 500.) The constitution of benzoïn and its nearest relatives are usually expressed as follows :

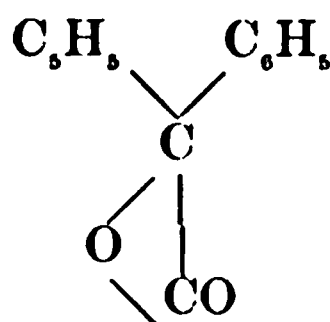


Benzoïn. Hydrobenzoïn, Desoxybenzoïn. Benzil.

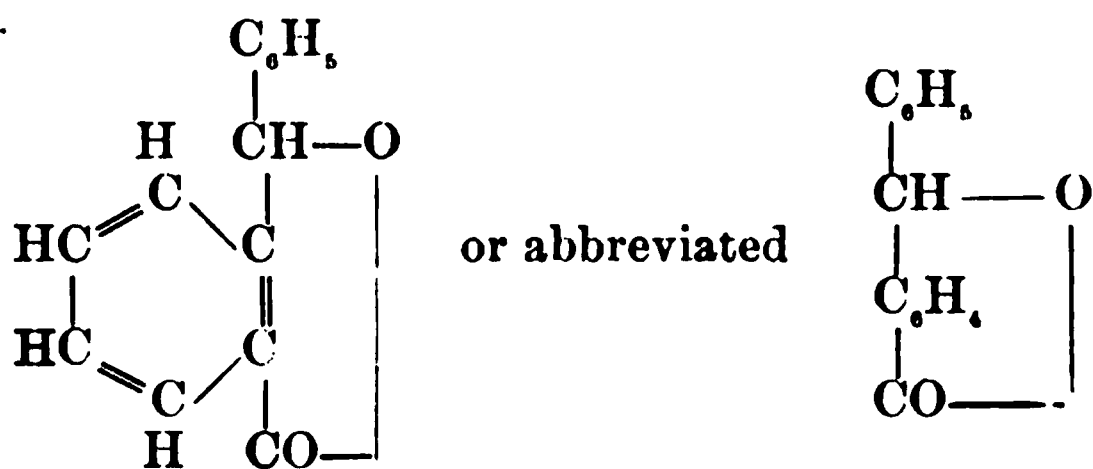
of these, however, only that of Desoxybenzoïn can be assumed to be correct with certainty. The authors state that the formula in common use for benzil is incorrect,  $\text{C}_6\text{H}_5\text{--CO--CO--C}_6\text{H}_5$ , as also the older formula :



Based on a number of experiments, the authors conclude about as follows : "By the investigations we think it proven that benzil contains but one carbonyl group. If we hence assume the formulæ just given for benzil to be incorrect we are still not capable of replacing either of them by a formula which we could rely upon. We would mention, however, that the formula formerly in use,

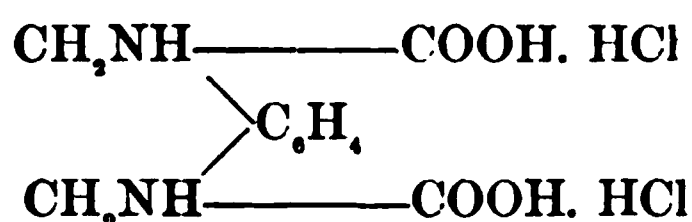


coincides with what we have found in reference to the position of the oxygen, and we will also not admit that our investigations could also be of considerable value if benzil was considered as a lactone, probably of the following formula :

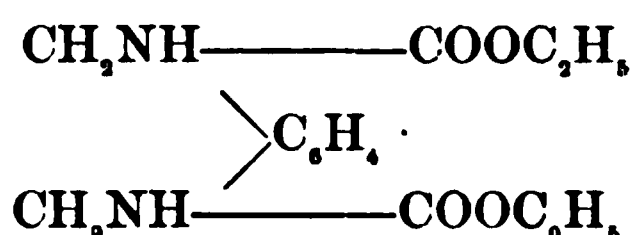


But against this formula also much can be said.

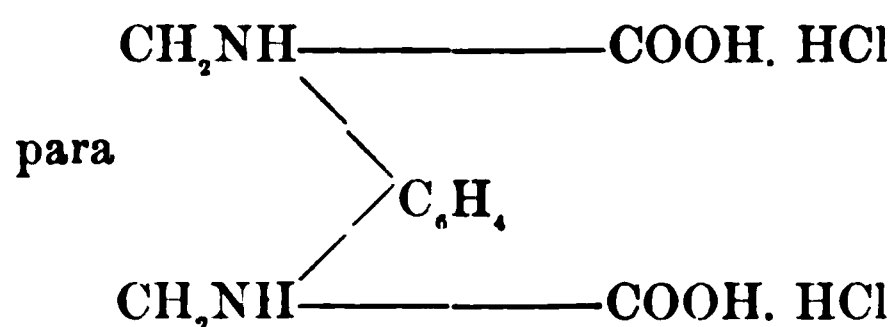
**Action Chloro-acetic Ether on Primary diamines.** J. ZIMMERMANN and M. KNYRIM. (Vol. XVI, p. 514.) By the action of chloroacetic-ether on meta-phenylen-diamine, meta-phenylen-diglycocoll-ether and phenylen-diamin hydrochloride are produced. If phenylendiglycocoll-ether is heated with strong hydrochloric acid with the reversed condenser or in sealed tubes at 100° crystals will separate on cooling. The analysis of these corresponds to the formula :



which is phenylen-diglycocoll hydrochloride. The para-phenylendiamin was also acted upon by chloroacetic-ether and the compound



was treated with hydrochloric acid in the same manner, giving



**On M-nitrophenyl Mustard Oil.** H. Standemann. (Vol. XVI., p. 548.) Meta-nitro-sulpho-carbanilid,  $\text{CS} \begin{smallmatrix} \text{NHC}_6\text{H}_4 \\ \text{NHC}_6\text{H}_4\text{NO}_2 \end{smallmatrix}$  was dissolved in hot acetic acid anhydride ; water was added, and the mixture boiled for a short time. An oil separates, which deposits crystals upon cooling. The reaction which by simple decomposition would yield phenyl-mustard oil, nitro-phenyl mustard oil, nitro-acetanilid or acetic acid and nitro-anilin, and acetanilid or anilin acetate respectively, seems to be a more complicated one in consequence of secondary reactions. If glacial acetic acid is added to the solution containing the deposited oil, crystals are obtained, more rapidly by the addition of little water. By pressing they are separated from adhering phenyl-mustard oil and are obtained pure by distilling with steam. They melt at 58°, and at that tempera-



ture emit the odor characteristic of all mustard oils. The analysis corresponds to the formula  $\text{CSNC}_6\text{H}_4\text{NO}_2$ .

**On Nitro-derivatives of Resorcin.** P. G. W. Typke. (Vol. XVI., 551). Diacetyl-resorcin was taken as a starting point. It was prepared by allowing 80 grms. of acetyl-chloride and 50 grms. of resorcin to act upon each other under a reversed condenser. The boiling point is  $275^\circ\text{C}$ .

Dinitro-resorcin was prepared by stirring diacetyl-resorcin into four or five times its volume of fuming, strongly cooled nitric acid, and then pouring the mixture upon ice. The nitro-compound separates as a white amorphous powder. This, after washing, pressing, extracting with alcohol and drying at  $100^\circ\text{C}$ ., was saponified by boiling with 30 per cent. hydrochloric acid under a reversed condenser. In order to separate this new compound from styphnic acid all was dissolved in a large quantity of hot water. On cooling the dinitro resorcin separated in fine needles. They melt at  $212.5^\circ\text{C}$ . They are best purified by crystallizing from boiling acetic ether. The analysis gives the formula  $\text{C}_6\text{H}_2(\text{NO}_2)_2(\text{OH})_2$ . Mono-brom-di-nitro-resorcin,  $\text{C}_6\text{HBr}(\text{NO}_2)_2(\text{OH})_2$ , is prepared from dinitro resorcin in glacial acetic acid solution and an excess of bromine.

Diamido-resorcin chloride  $\text{C}_6\text{H}_2(\text{OH})_2(\text{NH}_2)_2 \cdot 2\text{HCl}$  is prepared by reducing dinitro-resorcin at water-bath temperature by means of tin and hydrochloric acid. Oxidation probably gives rise to a di-imido-resorcin  $(\text{HO})_2\text{C}_6\text{H}_2(\text{NH})_2$ .

The constitution of di-nitro-resorcin could not yet be determined.

**On the Action of Bromine in Alkaline Solutions on Amines.** A. W. Hofmann. (Vol. XVI., p. 558.) If the hydrochloric acid solution of a primary monamine of the fatty series is treated with an alkaline solution of bromine, mono-alkylated brom-nitrogens are produced, which contain two atoms of bromine. The reaction was at first tried on the methyl and ethyl series and the compounds  $\text{CH}_3\text{NBr}_2$  and  $\text{C}_2\text{H}_5\text{NBr}_2$  were prepared and investigated; the experiments were then extended to amines of the propyl and pentyl series, two amylmines, the sextylamin and octylamin all have given analogous derivatives.

If an alkaline bromine solution is permitted to act upon a secondary amine of the fatty series, the primary alkylamin is formed by the separation of alkylenbromide if the secondary amin contains

two alkyl groups. But if a secondary amine in which a double group of atoms has taken the place of two hydrogen atoms of the ammonia, is treated with bromine, an alkylated brom-nitrogen is formed which contains only one atom of bromine. The formation of piperidin and coniïn derivatives has been studied.



The latter is especially interesting, and it was found by separation of hydrobromic acid, analogous bases are formed. This transformation may be perfected by acids or bases, and bases of the same composition, but of different properties, are produced.

If an acid is permitted to act upon the coniïn derivative  $\text{C}_8\text{H}_{16}\text{NBr}$  a base of the formula  $\text{C}_{18}\text{H}_{36}\text{N}$  is formed. It is very much like coniïn but boils at  $150^\circ\text{C}$ ., which is  $10^\circ$  less than coniïn. It is a tertiary while coniïn is a secondary base.

Alkalies convert the brom-coniïn into a base of the same composition but it is a secondary base,  $\text{C}_8\text{H}_{16}\text{NH}$ , and boils at  $173^\circ\text{C}$ . By reduction of these bases coniïn, and finally octylamin and octane are formed. These new compounds complete the desired series of amines with eight carbon atoms. There are known :

Collidin.....	$\text{C}_8\text{H}_{11}\text{N}$
Tropidin.....	$\text{C}_8\text{H}_{13}\text{N}$
New Coniïn Derivatives.....	$\text{C}_8\text{H}_{16}\text{N}$
Coniïn.....	$\text{C}_8\text{H}_{17}\text{N}$
Octylamin.....	$\text{C}_8\text{H}_{19}\text{N}$

The reactions with piperidine are more complicated. They have not been fully studied.

**An Easy Method of Preparing Paraffines.** B. Koehnlein. (Vol. XVI., p. 560.) In comparing the interchanges taking place between organic and inorganic halogen compounds an easy method was found by which the homologues of marsh gas, the hydrocarbons  $\text{C}_n\text{H}_{2n+2}$ , could be prepared.

2.5 grms. of pure chloride of aluminium free from chlorine and water, and 9.6 grms. of pure dry N-propyl-iodide in the proportion  $\text{AlCl}_3 + 3\text{C}_3\text{H}_7\text{I}$  were mixed in a tube. The air was exhausted, the tube drawn to a fine capillary point and heated several hours to  $130^\circ\text{--}140^\circ\text{C}$ . On cooling three substances could be distinguished. On opening the tube a gas was ejected with great force. In a

second experiment this gas was caught and proved to be pure propane.

2.40 grms.  $\text{AlCl}_3$  and 0.88 grms. of  $\text{C}_4\text{H}_9\text{I}$  heated in sealed tube to  $120^\circ\text{C}$  gave pure butane.

2.58 grms.  $\text{AlCl}_3$  and 9 grms.  $\text{C}_4\text{H}_9\text{I}$  heated five hours to  $140^\circ$ – $150^\circ\text{C}$ . gave pure ethane.

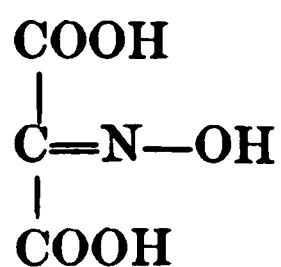
More investigations are in progress.

**Some Observations on Piperidin and Pyridin.** A. W. Hofmann. (Vol. XVI., p. 586.) The remarkable transformation of coniïn by the action of bromine (see above) gave cause to investigate the action of bromine on piperidin, and several experiments were successful, it being possible to prepare pyridin directly.

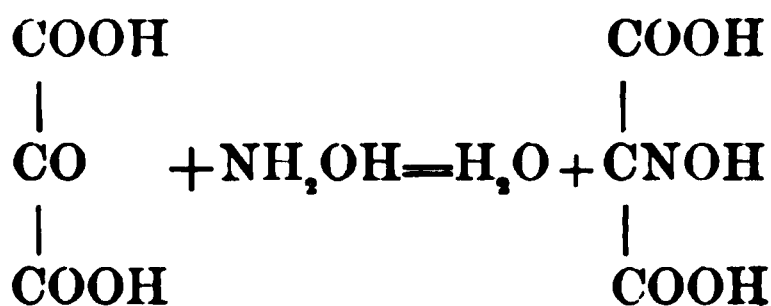
The aceto compound was used for a starting point. If a mixture of 1 mol. of the aceto compound is heated with 2 mol. bromine very gently, the temperature rises without more application of heat, and a colorless liquid distils over, leaving a syrupy residue. The distillate boils between  $80^\circ$  and  $200^\circ\text{C}$ ., and is a mixture of acetyl-bromide and its bromine substitution products.

If from the residue the bases are liberated by alkali and driven off with steam, an aqueous distillate smelling strongly of pyridin is found floating a basic oil layer carrying crystals. These when pressed and purified from alcohol have the melting point  $112^\circ\text{C}$ . They were found to be bibrom-pyridin. If pyridin was formed it was to be looked for in the aqueous distillate. Caustic potash separated an oil having the pungent odor of pyridin. The dried distillate boiled at  $110^\circ$ – $120^\circ$ , and was a mixture of pyridin and piperidin. They were separated by fixing the piperidin as an aceto compound. The base now distilled between  $112^\circ$  and  $116^\circ\text{C}$ ., and no longer became heated with acetic anhydride. It was almost pure pyridin.

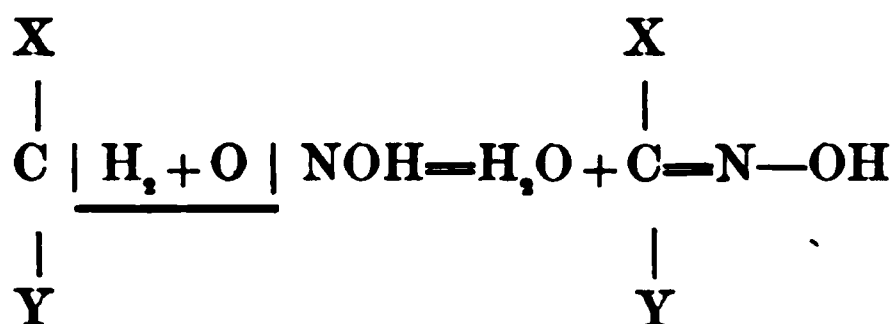
**On the Constitution of Nitroso-Malonic Acid.** VICTOR MEYER and A. D. MUELLER. (Vol. XVI. p. 608). It has been found by the authors that nitroso-malonic-acid is an iso-nitroso compound, having the formula :



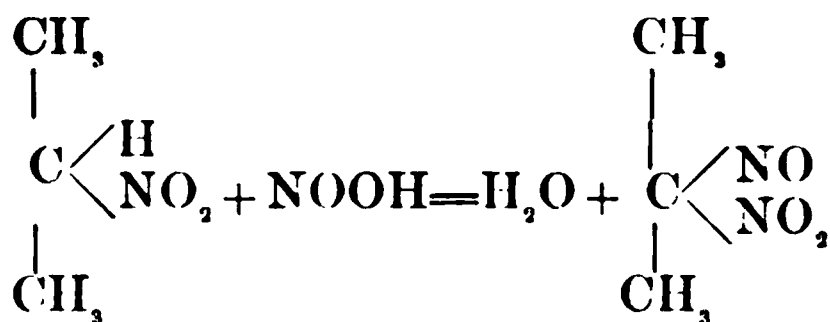
and that it could be formed from mesoxalic acid and hydroxylamin:



From this and other points the authors conclude that nitrogen compounds which are produced by the action of nitrous acid on the  $\text{CH}_2$  group are always iso nitroso-compounds. They are produced according to the equation :



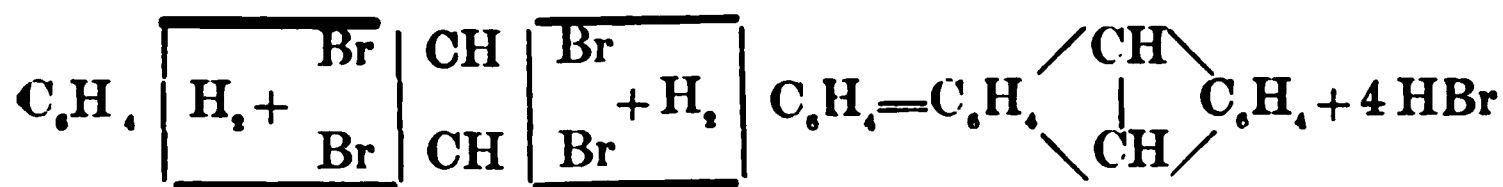
But if nitrogen compounds are formed by the action of nitrous acid on the group  $\text{CH}$ , when this is connected with a radical which can be easily removed, such as carboxyl, iso nitroso compounds are formed with the group  $\text{C} = \text{N} - \text{OH}$ ; but if nitrous acid acts upon  $\text{CH}$  groups which have radicals which are not easily removed, actual nitroso-compounds are formed containing the group  $\text{C} - \text{NO}$ . All aromatic nitroso compounds and the pseudo nitroles of the fatty series, the first representative of which is formed according to the equation :



belong to this category.

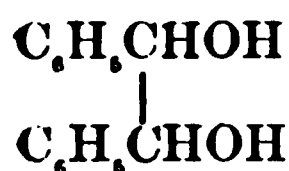
**A New Synthesis of Anthracene.** R. ANSCHUETZ AND F. ELTZBACHER. (Vol. XVI. p. 623.) According to the experience with aluminium chloride synthesis, it seemed probable that by the action of aluminium chloride on the two isomeric tetra-brom-ethanes—in benzol solution the term isomeric tetra-phenyl-ethanes would be formed. Owing to an uncertainty whether the tetraphenyl-ethane experimented upon was one and the same at all times, or whether there were two different ones, the authors undertook to

study the action of aluminium chloride upon a benzol solution of acetylentetrabromide. From the product of the reaction a substance is easily isolated which proved to be anthracene in place of the expected tetra-phenyl-ethane. The reaction could probably be assumed as follows :

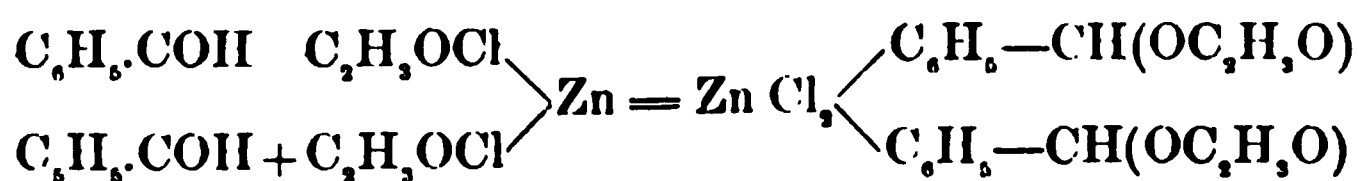
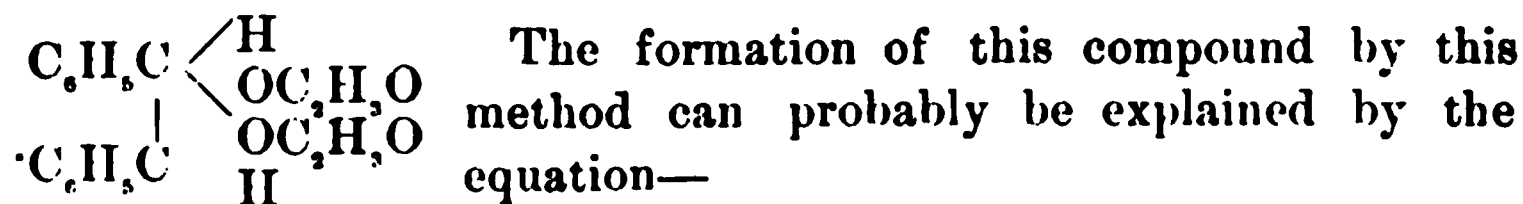


The investigations are to be continued.

**On the Action of Acetyl-Chloride on Benzaldehyde in Presence of Zinc Dust.** C. PAAL. (Vol. XVI. p. 636.) The compound formed was formerly given the formula  $\text{C}_{14}\text{H}_{12}\text{O}_2$ . When reduced with hydriodic acid and amorphous phosphorous it is converted into dibenzyl  $\text{C}_{14}\text{H}_{14}$  and distilled over zinc dust it forms stilben  $\text{C}_{14}\text{H}_{12}$ . Sodium amalgam was made to act upon an alcoholic solution of the substance at  $70^\circ$ – $80^\circ$ . The purified product melts at  $138^\circ\text{C}$ . and by analysis is found to be identical with hydrobenzoïn



The compound  $\text{C}_{14}\text{H}_{12}\text{O}_2$  reduced is decomposed, forming hydrobenzoïn and acetic acid. This makes the assumed formula very improbable, and pointed more to the hydrobenzoïn-diacetate.

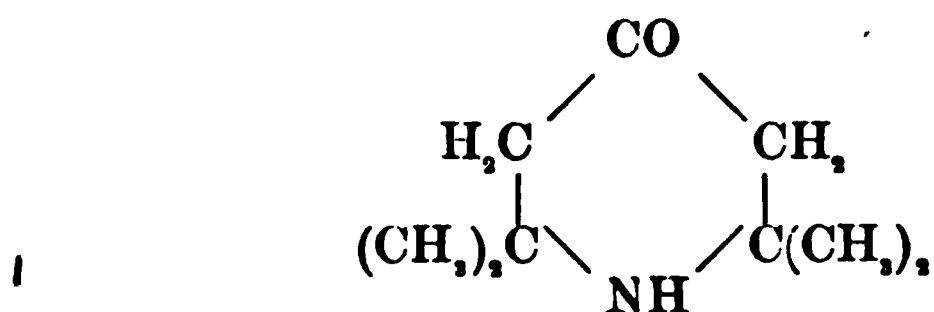


Some benzoic acid is also formed, showing that same oxidation also takes place.

**On the Oxidation of Piperidin.** C. SCHOTTE. (Vol. XVI. p. 643.) Piperidin when treated as its urethane with fuming nitric acid produces a saturated amido acid  $\text{C}_4\text{H}_8\text{O}_2\text{N}$ , which differs from

the coniïn acid by  $C_2H_5$  in the same manner as piperidin differs from coniïn by minus the group  $C_2H_5$ . Intermediate products of oxidation are formed, which are also described.

**Note on Triacetonalkylamin.** EMIL FISCHER. (Vol. XVI. p. 649.) Heintz gives it the formula—



If this is correct the alkylamin produced by reduction must be an hydroxylated tetramethylpiperidin. In order to prove this it was tried by abstracting water from triacetonalkylamin to convert it into a base of the piperidin series containing no oxygen. The reaction with a mixture of penta-chloride of phosphorous and oxychloride of phosphorous is very complicated. The reaction with conc. sulphuric acid is more simple. The free base forms a well crystallized hydrate with water. The analysis of the hydrochloride gave figures corresponding approximately to the formula  $C_{11}H_{17}N.HCl$ .

M. & LINING  $\frac{1}{2}$  cc OF  
P. M. & P. A

10 cc.	10 cc.	4th 10 cc.	5th
NH <sub>3</sub>	$\frac{1}{2}$ cc NH <sub>3</sub>	$\frac{1}{2}$ cc NH <sub>3</sub>	

002	001	.....	
001	001	.....	
001	001	.....	

Nil	020	.0005	....
005	010	Nil	....
010	005	.0005	....

001	001	Nil	....
001	001	.0010	....
001	001	.0005	....

018	012	.004	.C
011	014	.009	.C
023	009	.004	.C

001	004	.0010	....
001	001	Nil	....
001	001	.0005	....

004	Nil	Nil	....
002	Nil	Nil	....
002	001	Nil	....

09	010	.....	
10	010	.....	
05	005	.....	

10	005	Nil	....
10	Nil	Nil	....
05	005	.0005	....

11	Nil	.....	
05	Nil	.....	
05	005	.....	

1	Nil	.....	
1	005	.....	
01	Nil	.....	

1	005	.....	
11	005	.....	
1	005	.....	

1	Nil	.....	
2	001	.....	
2	Nil	.....	

2	010	Nil	.....
2	005	Nil	.....
2	Nil	Nil	.....

3	005	Nil	.....
3	005	Nil	.....
3	010	Nil	.....





# THE ATOMATION OF OXYGEN.

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BY ALBERT R. LEEDS, PH. D.

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## FIRST MEMOIR.

The readers of this journal will have noted that ever since the publication of my earliest paper, bearing upon the subject of the Atomation of Oxygen, and entitled "Upon Ammonium Nitrite and upon the By-products obtained in the Ozonation of Air by Moist Phosphorus," Vol. I., p. 145, I have taken the ground that the essential feature in oxidation-processes was the splitting of the oxygen molecule, and the setting free of atomic oxygen, or oxygen in the state of free atoms (sometimes called active or nascent oxygen). I have preferred to employ the term atomic, and the phrase atomation of oxygen, rather than the terms *active* or *nascent*, because ozone is eminently active oxygen, while at the same time it consists of atoms in a combined condition.

The experimental proof of the proposition, that the oxygen must be brought into the atomic condition before it can re-combine to form ozone, hydrogen peroxide, ammonium nitrite, carbon dioxide, etc., is so fully given in the recent paper, "Conversion of Carbon Monoxide into Carbon Dioxide by Active (i.e., Nascent) Oxygen," that it is needless to rehearse it.

My present object is to present two hypotheses differing from the foregoing, the first by Hoppe-Seyler, the second by M. Traube, an animated controversy concerning which has been carried on for many months.

The first publication by Hoppe-Seyler in the Berlin Berichte upon this topic was in 1879, Vol. XII., p. 1551, "Upon the excitement of oxygen by nascent hydrogen." He begins by stating that every investigation undertaken with the object of explaining the life-processes in animals and plants, necessitates the assumption of some adequate source of the activation of oxygen within the organism. He deduces conclusions upon this point from certain phenomena attendant upon putrefaction, and states that the formation

of free hydrogen occurs under these conditions only when oxygen is absent. On the other hand, when oxygen is admitted to putrefying liquids, the nascent oxygen is not only oxidized, but energetic processes of oxidation are set up. The simplest explanation of these facts, according to Hoppe-Seyler, is the assumption that the nascent hydrogen, whilst it itself enters into combination with oxygen, at the same time brings to pass the activation of one or more atoms of oxygen. To test the validity of this hypothesis, Seyler proposes the use of hydrogenized palladium. "From Graham's experiments," he says, "it is known that palladium at ordinary temperatures takes up no oxygen, and one can readily satisfy himself that the ignited foil when placed in a confined volume of oxygen does not change. The oxygen, therefore, is not made active by the metal, whilst foil saturated with hydrogen and brought into oxygen, not only quickly forms water, but likewise is in a condition adequate to bring to pass the most energetic processes of oxidation." As proof, he instances the oxidation of indigo by these means, the decomposition of potassium iodide, the conversion of ammonia into ammonium nitrite, and the production of phenol from benzol.

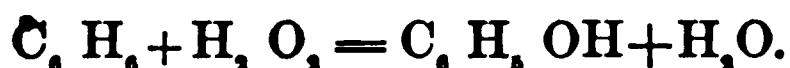
Further on, he describes the products of oxidation of petroleum-ether by sodium in contact with moist air, but does not decide whether these oxidation-processes are due to oxygen rendered atomic by direct action of the sodium, or by the hydrogen first set free and afterwards decomposing the oxygen molecule.

In this publication no mention is made of the formation of hydrogen peroxide, nor is the antecedent probability of its formation on hypothetical grounds alluded to.

On meeting with this research of Hoppe-Seyler, it appeared to me that there was no reason why the atomation of oxygen by palladium-hydrogen should not be in obedience to the same law, as its atomation by phosphorus. Nor any reason why the same secondary products should not be necessarily formed, as resultants of the recombination of this atomic oxygen with itself or with oxygen to form ozone, with water to form hydrogen peroxide, and with nitrogen and the elements of water to form ammonium nitrite and nitrate. The probability was rendered stronger, because I had performed the experiment, before encountering these results detailed by Hoppe-Seyler, of submitting benzol to oxygen in presence of moist phosphorus, and found that, under these circumstances, the benzol was converted into phenol.

On trying the experiment with hydrogenized palladium in pres-

ence of air and water, the formation of hydrogen peroxide was demonstrated (Ber. Bericht. XIV., 976). Furthermore, since the formation of phenol from benzol could not be accounted for by direct oxidation with atomic oxygen, and since I had shown that ozone decomposes benzol entirely into oxalic, formic and acetic acids, together with a small amount of some black, amorphous substance (ib. p. 975), the formation of phenol in Hoppe-Seyler's experiments could be explained only on the supposition of the intermediate production of hydrogen peroxide. This supposition proved correct, and an experiment in which benzol was heated directly with hydrogen peroxide, yielded phenol according to the equation :—



Of the two other by-products (ozone and ammonium nitrite) I looked for the former only, because I was satisfied with the demonstration of the production of ammonium nitrite given by Hoppe-Seyler (loc. cit.) This last point, however, has been emphatically denied by Traube (Ber. Bericht. XVI., 1206) on the strength of obtaining a negative result, thereby inciting Hoppe-Seyler to bring forward the following experiments (Ib. 1923.)

“All the glass apparatus and caoutchouc tubing, just previous to the beginning of the experiment, together with the hydrogenized palladium, were cleansed with water, which had been distilled with pure sulphuric acid and afterwards with a little pure soda. From the glass vessel, in which was the wet foil (91 square centimetres in area and 13.2 gm. in weight), the air went through a wash-bottle with some pure water, then through caustic soda, and finally through sulphuric acid. The foil was allowed to remain for three days in the vessel, and a small fresh portion of air was drawn through the apparatus very two or three hours. At the close of the experiment the foil and the interior of the vessel were washed out with a couple of cubic centimetres of pure water, two or three drops of pure caustic soda added and the mixture evaporated with boiling to a small volume. After cooling, it was acidulated with pure sulphuric acid, and tested with (1) potassium iodide starch paste; (2) with sulphanilic acid and naphthylamine sulphate; (3) with metadiamido-benzole; (4) with sulphindigotic acid; (5) with ferrous sulphate and sulphuric acid. In like manner, the water in the wash-bottles through which the air after passing from the glass vessel was drawn, was tested.” In both liquids, the sulphindigotic acid

and the ferrous sulphate with sulphuric acid did not give reactions, the other three test-liquids did.

I have been thus particular to quote these elaborate experiments, because the establishment of the fact of the production of nitrous acid under the conditions studied, is of so great theoretical importance, and because the cumulative evidence of the certainty of this fact, can best be appreciated by a critical examination of the above detailed account of Hoppe-Seyler's labors.

The discovery of the formation of hydrogen peroxide has not only been admitted on all hands, but apparently has three claimants. For whilst Traube brought it forward as a new observation of his own (Ber. Bericht. XVI. 222), and placed great stress upon it in support of his hypothesis, later on he calls attention to his mistake, and credits me with the observation (*ib.* 2429). In the same way, he states that Hoppe-Seyler had overlooked the production of hydrogen peroxide in his article upon the activation of oxygen, written in 1879 (Ber. Bericht. XII. 1551). But Baumann states (Ber. Bericht. XVI., 2149) that Hoppe-Seyler first noted the phenomenon (Zeitschr f. physiol. Chem. II., 25), and that, therefore, when he himself observed it in the contact of hydrogenized palladium with water and air, he regarded it as no new thing. (Zeitschrift, f. physiol. Chem., V., 248). Not having access to the Zeitschrift referred to, I am unable to form an opinion as to whether Traube or Baumann is right in this particular. [See Appendix to this article.]

The third body, the ozone, which, according to my interpretation of these phenomena, is always to be looked for, was sought in my own experiment in the atmosphere in the bottle over the hydrogenized palladium. But although only a negative result was obtained, as, in fact, I anticipated, in view of the very small amount of oxidation possible by a small piece of hydrogenized palladium (Bericht. XIV., 978), yet I see no more *a priori* reason militating against the probability of the formation of ozone by the hydrogenized palladium, than for the production of hydrogen peroxide or ammonium nitrite.

Subsequent to the work above alluded to, a long series of articles upon the "Activation of Oxygen" has been published by M. Traube. In the earliest of these (Ber. Bericht. XV., 222), Traube lays down the following propositions :

I.—"Palladium-hydrogen gives, when shaken with water and air, immediately and in large quantity hydrogen peroxide. The forma-

tion of hydrogen peroxide under these circumstances has been hitherto overlooked."

It has been already mentioned that Traube himself corrects the latter part of the statement, and credits me with having first noted the reaction. I do not know where Traube has shown by quantitative determination that hydrogen peroxide is formed in notable amount. In one of my own experiments with a piece of hydrogen palladium weighing 10 grammes, 0.67 mgrm. of hydrogen peroxide was obtained. Hoppe-Seyler, in a recent publication (Bericht. XVI., p. 1920), obtains a similar result. He states that with palladium, hydrogenized to its maximum, whether the contact with water and air is continued only a few minutes or many hours, the percentage of  $H_2O_2$  does not increase beyond 2 mgrm. in a liter. The highest percentage obtained was 6 mgrm. per liter."

II.—"The oxidations effected by palladium-hydrogen, in presence of air and water, do not proceed from it directly, but entirely from the resultant hydrogen peroxide."

III.—"There is one case only, so far as my present experience goes, in which the oxidation-processes effected by palladium-hydrogen, in presence of air and water, are different from those of hydrogen peroxide. Whilst the last does not turn potassium-iodide-starch blue, rapid bluing occurs by palladium-hydrogen and oxygen. This action is due to the palladium playing the part of a carrier of oxygen from the hydrogen peroxide to the potassium iodide."

IV. "In opposition to the view of Hoppe-Seyler, my investigations have shown that nascent hydrogen is not able to activate oxygen by splitting of its molecule."

V. "The frequent production of  $H_2O_2$  by oxidation-processes, is not proof of the contemporaneous presence of active oxygen, since this  $H_2O_2$ , as I have shown by direct experiments and contrary to former assumption, never arises by oxidation of water by means of active oxygen. In these oxidation-processes it is formed by a mode of reduction."

These four last propositions, have provoked a controversy, which has run through many numbers of the *Berichte*, and has become so involved and so voluminous that I fancy but few chemists have found leisure and inclination to follow it.

And yet to chemists in general it should have great interest, because the discussion involves the endeavor to demonstrate, by critical experiments, the nature of those obscure chemical reactions involved in the so-called nascent state, catalysis, oxygen-carrying, etc.

To physiological chemists its interest should be no less. On the one hand, Hoppe-Seyler thinks that he has demonstrated that it is hydrogen which possesses in the nascent state the power of producing in contact with ordinary oxygen, the most energetic oxidations, water at the same time being formed. "These facts, he remarks, are of the greatest importance in physiology, because for the first time it has been shown from the properties of chemical substances that the energetic oxidations stand in well ascertained dependence upon other processes, by which bodies are formed that act upon oxygen in the same way as hydrogen when in the nascent state." Traube, on the other hand, ascribes the activation of oxygen in the organism to oxygen-carriers. By oxygen-carriers he means bodies like platinum, which, according to the explanation of De la Rive, have the property of readily yielding the oxygen which they have taken up to other bodies, and then forthwith as quickly taking up new oxygen again.

. I propose to consider these several propositions and present the arguments and experiments which have been brought forward both pro and con. Inasmuch, however, as besides the contradictory results obtained by the contending parties in the performance of the same experiments, there is a different mode of interpreting the significance of these results, there will be no use in weighing the value of the experimental evidence until some mode of interpretation is agreed upon which will be impartially applied in every case.

#### INTERPRETATION OF RESULTS.

And 1st. It must be agreed I think on all hands, that the existence of atomic oxygen, as a substance distinct from, and antecedent to the production of ozone, is a demonstrated fact. The demonstration consists in the proof that this atomic oxygen will effect oxidations which ozone will not, as in the oxidation of carbon monoxide to dioxide, at common temperatures. And the converse of this proposition is likewise true, which is, that when ordinary oxygen is brought into a condition to effect the oxidation of carbon monoxide to carbon dioxide atomic oxygen must be present, and the substance which is capable by its presence of effecting this oxidation must have the power of splitting the oxygen molecule, that is, of setting free its constituent atoms.

The original experiment upon which the first proposition was founded was that of the oxidation of carbon monoxide in the passage of air over moist phosphorus. Although both ozone and hydro-

gen peroxide are formed in the course of this experiment, yet as neither of these bodies will oxidize the monoxide, the necessity of the existence of atomic oxygen to account for the fact, is admitted. Subsequent to this observation, Baumann noted that palladium-hydrogen had the same property as phosphorus, and that in its presence moist air could raise the monoxide to dioxide (quoted in Ber. Bericht., XVI. 123). This result prompted Traube to ascertain whether or no ignited palladium placed in dilute hydrogen peroxide and agitated in contact with carbon monoxide out of contact with air, would produce carbon dioxide (Bericht, XVI. 126). He obtained an affirmative result, and this result is confirmed by Baumann (*ib.* 2150). But their modes of interpretation are different. Traube reasons that inasmuch as air was absent no atomic oxygen could have been formed, but that the palladium by catalytic action caused the hydrogen peroxide to effect an oxidation which alone it could not have effected.

Whilst admitting and confirming Traube's result, for Baumann agrees with Traube in finding that ignited palladium in contact with  $\text{H}_2\text{O}_2$  and CO produces much more  $\text{CO}_2$  than palladium-hydrogen when agitated with CO and water, Baumann's explanation is different. There appears to have been a tacit admission on his part that the formation of  $\text{H}_2\text{O}_2$  must occur as an intermediate step when the palladium-hydrogen is agitated in contact with water and CO out of contact with air. Further, that it is possible that this  $\text{H}_2\text{O}_2$  is then decomposed by the palladium, ozone being formed. (He might have added, atomic oxygen is likewise formed.) And although this formation of ozone by decomposition of  $\text{H}_2\text{O}_2$  with palladium has not been observed, yet he regards it as eminently probable, inasmuch as when  $\text{H}_2\text{O}_2$  is decomposed by sulphuric acid (this fact was originally noted by Riche (Bull. Soc. Chem., 1860, 178), ozone is given off. Having repeated this experiment of Riche (J. Amer. Chem. Soc., I., 442), I can confirm his statement, the liberated ozone being so powerful as to blacken silver foil. But Baumann appears to have gone further and to have subjected carbon monoxide to the action of the evolved gas, since he states (without giving the particulars of the experiment) that the carbon monoxide was more powerfully oxidized by this gas than by any of the other methods. This being the case, as Baumann justly observes, the decomposition of the  $\text{H}_2\text{O}_2$  by sulphuric acid must be attended by the evolution of atomic oxygen as well as ozone.

Furthermore, Baumann tried the experiment of passing moist air



over palladium-hydrogen to see whether the water condensed after its passage contained  $\text{H}_2\text{O}_2$ . The result was negative. But inasmuch as moist air and CO passed over dry palladium-hydrogen had yielded  $\text{CO}_2$ , Baumann thinks that the negative result might be accounted for on the ground that the atomic oxygen could have produced  $\text{CO}_2$  under circumstances, under which the oxidation of water to  $\text{H}_2\text{O}_2$  was not demonstrable. Since, later on, Baumann says that he agrees with me in the interpretation I have given of these phenomena, I think he would further assent to the following as a more explicit formulation of his own remarks and observations, viz. :—

That inasmuch as carbon monoxide is oxidized under the conditions stated, the palladium must have effected the atomation of the oxygen molecule. The oxygen set free from the  $\text{H}_2\text{O}_2$ , which, when spontaneously liberated will not oxidize CO to  $\text{CO}_2$ , will bring about this oxidation in the presence of palladium. In other words, the so-called catalytic actions of palladium, platinum, and metals of that group are due to atomation.

Since these metals do not combine with the oxygen under the conditions named, they are in no proper sense *carriers of oxygen*. They do not carry oxygen, but, on the contrary, decompose it, bringing it into the atomic condition, and thus effect their so-called catalytic actions.

#### EXPERIMENTAL PROOFS.

In his first paper (Bericht. XV., 664). Traube brought forward the deportment of zinc when shaken in contact with water and air, both in neutral and acid solution, in support of his hypothesis ; also that of copper in presence of sulphuric acid.

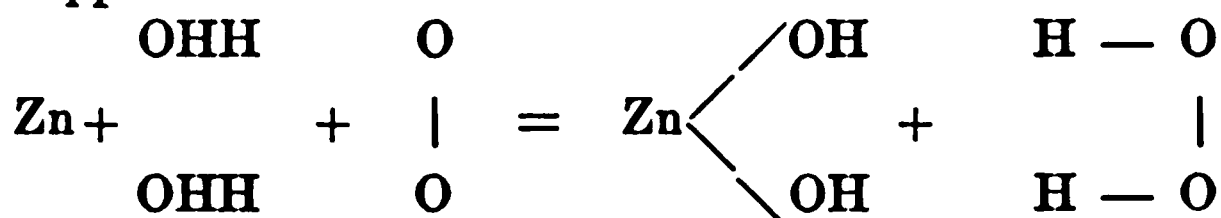
But in the mode of statement, the actual conduct of the experiments and the inductions based thereon, these proofs are of the most inconclusive character.

Traube begins by stating that in case the oxidation of zinc, by shaking with air and water, produces atomic oxygen, then an oxidizable body present at the same time must be rapidly oxidized. But he finds that whether pure zinc or very dilute sulphuric acid is employed, such oxidation is not produced, although in both cases abundance of  $\text{H}_2\text{O}_2$  is formed.

Traube places great emphasis on the fact that pure zinc out of contact with air does not decompose water and set free its hydrogen. Therefore, that what the affinity of the zinc alone for the oxygen of the water will not effect, this affinity, aided by the affinity of a whole molecule of atmospheric oxygen, will succeed in



doing ; that is, the two together will split up the molecule of water. The supposed reaction is as follows :—



According to Bonsdorff and Boutigny (Gmelin-Kraut, 3d vol., 1st part, p. 5), when zinc is inclosed in glass tubes with water from which the air has been expelled by boiling, it remains unaffected for years. According to J. Davy (*ibid.*) it does not decompose pure water even on boiling, and even in contact with copper it does not appreciably decompose pure water.

In repeating these experiments I have obtained different results. The purest zinc which I could procure contained lead, iron and carbon in minute amounts, and consequently the deportment of absolutely pure zinc with pure water is not determined by my own trial. Forty grammes of this zinc in extremely thin fine turnings were introduced into the upper portion of a eudiometer tube completely filled with redistilled water free from ammonia. The tube was surrounded by a water bath and maintained at a temperature of 70° for seven hours, during the whole of which time it was connected with an air-pump and a vacuum maintained. The open end of the eudiometer was then plunged into a vessel filled with mercury, and the apparatus allowed to stand from the middle of June to the middle of September. Between the upper surface of the mercury and the zinc in the eudiometer there intervened a space of 8 centimeters filled with water, so that contact between the two metals could not occur. During the whole of this long interval bubbles of gas came off slowly, and finally amounted to a volume of 15.95 cubic centimeters, reduced to 0° and 760 m.m. After the introduction of a suitable excess of oxygen and explosion by the electric spark, a reduction of the measurements showed that the 15.95 cubic centimeters of gas collected during the course of three months consisted of hydrogen. The zinc was very faintly tarnished, appearing somewhat less bright, and of a slightly different color from fresh surfaces of other portions of the same zinc, but no oxide was visible.

Now, it is a well-known fact that zinc, especially when in fine powder, may be employed to bleach a solution of indigo. In this case the indigo blue ( $\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_2$ ) is converted into reduced or white indigo ( $\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_2$ ) by the fixation of nascent hydrogen.

Two interpretations of this phenomenon are possible :—1st.

That the hydrogen liberated from water by ordinary zinc in the manner above described, small in amount though it may be, is adequate in amount, when a large surface of zinc dust is employed, to reduce the indigo. 2d. That soluble indigo or sulphindigotic acid, being acid in its reaction, liberates the nascent hydrogen on contact with zinc, which brings about its reduction. The latter is the correct explanation. For when sulphindylate of potash is employed, the salt being made as nearly neutral as possible, it is found that zinc does not act as a reducing agent, but as a powerful oxidizing agent. Under these circumstances, the blue solution is bleached, it is true, but not by the reduction of the indigo, since the solution retains a yellow tint in contact with the air, and contains no white indigo, the indigo having undergone oxidation. When large amounts of indigo carmine are employed this complete oxidation and decoloration are somewhat difficult to obtain, because of the energy with which fine shavings of zinc are themselves oxidized and the great amounts of zinc hydrate which are formed. On shaking up a liter bottle containing a hundred grms. of zinc in fine shavings, with 100 cc. of indigo carmine solution, the contents of the flask become at once bluish-gray from admixture of suspended zinc hydrate. In this first bottle, which now contains oxidized zinc, complete decoloration is hard to effect, but on filtering off the solution into a bottle containing fresh zinc, bleaching occurs immediately.

My own observations, therefore, are directly opposed to those of Traube, who stated that zinc would not bleach solution of indigo. Moreover, inasmuch as the indigo in my experiments was oxidized and not reduced, they show that zinc shaken up in contact with water and air brings about phenomena of energetic oxidation.

In the second place, hydrogen peroxide is formed under the same conditions. As stated by Traube, the reaction for the peroxide entirely disappears in case the water, after brisk agitation with the zinc, is allowed to remain in contact with it. And inasmuch as the reaction with potassium iodide or with potassium iodide and ferrous sulphate is affected by substances other than  $H_2O_2$ , the author did not rely upon these tests in proving qualitatively the presence of the latter body. Instead of so doing freshly-prepared cold malt extract was used. Upon this was floated freshly-made tincture of guaiacum, and it was noted whether any blue color was developed along the surface of contact of the two liquids. If not, the water decanted from the surface of the zinc was poured down the

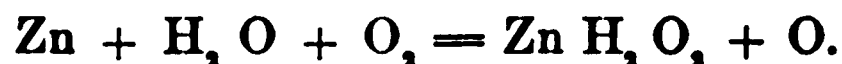
side of the test-tubes, so as to flow under the guaiacum tincture and above the malt extract. Only when these precautions were observed was the development of a blue color regarded as a satisfactory indication of the formation of hydrogen peroxide.

The oxidation of indigo by zinc in neutral solution would lead us to anticipate the decomposition of potassium iodide under the same circumstances. This anticipation is verified by experiment in case starch is added to the potassium iodide and the mixture then shaken up with the zinc. Under these circumstances iodide of starch is formed, whilst it is not formed in case potassium iodide solution alone is agitated with zinc, the liquid poured off, and then starch added.

*Nitrous Acid.*—When redistilled ammonia-free water, which failed to give any coloration with sulphanilic acid, was agitated with zinc, the decanted liquid gave a decided reaction for nitrous acid. But when ordinary distilled water, which itself gave a faint coloration with sulphanilic acid, was shaken with zinc, the decanted liquid gave no reaction for nitrous acid, and remained entirely colorless. These reactions were explained by those occurring when a solution of potassium nitrite, standardized for purposes of water analysis, was treated with hydrogen peroxide. After forty-eight hours it failed any longer to give the nitrous reaction, the acid present having undergone oxidation to nitrate.

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To summarize the results detailed above, it may be stated that there is the same evidence of the production of atomic oxygen, when zinc is agitated in contact with water and air, as there is of the production of atomic oxygen when moist air is passed over phosphorus. Moreover, that this atomic oxygen arises from the splitting of the oxygen molecule by the zinc directly, and not by means of hydrogen resulting from any intermediate process. In other words, zinc reduces the oxygen, being rapidly converted in presence of water into hydrate, and setting atomic oxygen free, the equations being



It is this atomic oxygen, which the author supposes to be the efficient agent in the subsequent formation of hydrogen peroxide and nitrous acid, and the origin of the energetic oxidation of the neutral indigo solution and of the decomposition of the potassium iodide in presence of starch.

[To be continued.]

## APPENDIX.

The principal portion of this First Memoir was read at the June meeting of the Society, but its publication was delayed by the pressure of other duties subsequently devolving on the author.

Since the article was written I have had the pleasure of receiving letters both from Prof. Hoppe-Seyler and from Prof. Baumann, the former dated November 17, 1883, and the latter, December 10th. Prof. Hoppe-Seyler has likewise kindly sent me reprints of his papers in the *Zeitsch. für Physiol. Chemie*, bearing upon the matter referred to, and these, together with an extract from his letter, which I shall take the liberty of quoting, will best illustrate the history of the subject. Prof. Hoppe-Seyler says, \* \* \* \* “Both of us nearly at the same time and quite independently of one another, have investigated the properties of active oxygen, and in general with the same results. But it would appear from a passage in your memoir, ‘The conversion of carbon monoxide into carbon dioxide by active (*i. e.* nascent) oxygen,’ that my earlier publications have remained unknown to me. You there remark, ‘According to these views the oxidizing effect of palladium hydrogen, as noted by Hoppe-Seyler at a late period in the history of these researches,’ etc.

“My earliest observations upon the activating agency of hydrogen when in *stat. nasc.* were published as far back as 1876, and in 1877 were further elucidated in my *Handb. der physiol. Chemie*. The powerfully oxidizing action of H in *stat. nasc.* in the presence of oxygen when palladium-hydrogen is employed, I had already intimated in a preliminary note dated February 21, 1878, and shortly after this it was fully described in the spring of 1878 in *Zeits. für physiol. Chemie*, Bd. II., p. 22. Some further observations caused me to publish a short summary of the results so far obtained, in the *Berichte*. The formation of  $H_2O_2$  in certain reactions I had already noted in the beginning of my investigations. but the oxidations which are effected by the agency of H in *stat. nasc.* are more energetic than those produced by  $H_2O_2$ .”

The statement alluded to by Prof. Hoppe-Seyler (*Zeitsch. für physiol. Chemie*, Bd. II., p. 22) is as follows: “By far the most interesting reduction which active hydrogen is capable of effecting, is that of free indifferent oxygen, attended with the formation of water, whether it results in this case that hereby ——— OH or a combination  $HO-O$  ——— or  $H_2O + -O-$  is formed. This is the

origin of powerful oxidations, which indifferent oxygen is not capable of effecting."

Pages 24 and 25, of the same article contains the statement alluded to by Baumann, as showing that I was anticipated by Hoppe-Seyler, in stating that  $H_2O_2$  is a product consequent upon the formation of atomic oxygen in presence of water. It is as follows :—

"Like the liberated hydrogen atom, so also the O-atom cannot remain free, but in case no other oxidizable substances are present it forms with water or indifferent oxygen, either  $H_2O_2$  or  $O_3$ ."

"The occurrence of nitrous acid in the combustion of hydrogen with air has given occasion to Bunsen (*Gasometr. Meth.* 1857, p. 65) to set forth appropriate precautions in gas analysis. Recently Zöller and Grete (*Ber. deutsch. Chem. Gesell.* X, 2144), have recognized the occurrence of small but clearly distinguishable quantities of  $NH_4NO_3$  in the combustion of pure hydrogen gas in pure atmospheric air."

"The action of hydrogen upon indifferent oxygen is the same therefore as the action of many other substances, which possess a very strong affinity to oxygen, such strongly reducing substances, for example, as phosphorus and magnesium."

"It has been lately found by Kämmerer (*Berichte* X, 1684) that in the combustion of magnesium in air, nitrous acid is formed. In the slow combustion of P at least one atom of oxygen becomes active for every molecule  $P_4$ ."



"The discovery of Schönbein that powdered zinc and iron, when shaken up with air and water, give rise to the formation of  $H_2O_2$ , can likewise be explained in hardly any other manner than as a result of a reduction of the indifferent oxygen."

"None of these processes, however, shows simply and clearly like the decomposition of palladium-hydrogen does, the reduction and oxidation effected by means of such decomposition."

I am very glad to yield to Prof. Hoppe-Seyler the merit of having investigated the phenomena consequent upon the evolution of atomic oxygen in a large number of cases, and more especially in that arising from the contact of palladium-hydrogen in contact with air and water. And the difference between his labors and my own appears to consist in the fact that whilst Hoppe-Seyler was engaged upon the demonstration of the formation of atomic

oxygen itself as the main topic of inquiry, I was more intent upon establishing the thesis that whensoever atomic oxygen is produced in the presence of air and water, ozone, hydrogen peroxide and ammonium nitrite are necessarily and simultaneously formed as secondary products. This hypothesis led me to anticipate, in entire ignorance of Hoppe-Seyler's labors, the formation of  $H_2O_2$ , when palladium-hydrogen is agitated in contact with water and air, to establish the fact of its formation by experimental proof, and to determine quantitatively its amount. I think it not invidious, nor an undue detracting from the merits of Hoppe-Seyler's discoveries, to claim for myself priority in the two latter points.

STEVENS INSTITUTE OF TECHNOLOGY,

December 15, 1883.

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## ON A NEW OVERFLOW PIPETTE.

GEO. S. EYSTER, PH.D.

The well known Overflow Pipettes of Gay-Lussac and Stas, whilst adapted for the accurate measurement and delicacy of a given volume of a solution, are not so fit for the use of corrosive fluids, and are not easily extemporized from materials commonly found in a laboratory.

The following pipette is especially adapted for use in alkalimetry, and for the employment of such corrosive solutions as are used in the "copper" method of estimating sugar (Fehling's, &c.)

An ordinary pipette, graduated to deliver a definite volume, is clamped in a *reverse* position; that is with the mark *down*, and the jet *up*. A short piece of rubber tube connects the end that is now the lowest end with one arm of a glass T tube, the other arm of which is provided with the usual Mohr burette tip; or with a glass bead cock.

This glass bead cock is simply a short piece of glass rod, somewhat larger than the bore of the rubber tube, fused into a bead. This bead inserted into the rubber tube effectually stops the flow. When it is desired to open the cock, a slight pinch of the tube over the place where the bead is concealed, will open a channel for the passage of the solution. The flow can be regulated with the greatest nicety. This piece of apparatus is *old*, but does not seem to be as generally known as it should be.

To the lateral arm is attached the rubber tube and pinch cock which supplies the pipette with the solution from the reservoir. The reservoir is either a common bottle, provided with a syphon, or an aspirator bottle. The higher the level of the reservoir above the tip of the pipette, the quicker the pipette will be filled.

In place of the glass cups used in the Stas pipette, I make use of the following arrangement to catch the overflow from the open end.

A large test tube is fitted with a cork with two holes. Through one the jet of the pipette is passed. One arm of a rather wide bent glass tube is inserted in the other hole. This is the drip tube, and is provided with a rubber tube to carry away the excess of solution.

It is easy to see the mode of action. When the pinch cock leading to the reservoir is opened, the solution enters and fills the pipette; the air escapes through the drip tube. When the solution is to be delivered, the lower cock is opened, and the fluid run out until the mark is reached; the air meanwhile enters by the drip tube, and for this reason care must be taken to have the drip tube as wide as possible, and not to allow the rubber drainage tube to dip beneath the fluid in the vessel it connects with.

The error due to reading the meniscus in a reverse position is easily corrected, and in many cases will be eliminated in standardizing the solutions.

I give the simplest form, and one readily made from easily available materials.

If one is somewhat expert in glass blowing, the end of the large test tube (or the wide tube used instead) can be drawn out and bent, so that its extremity can be left open for the admission of air. There is some advantage in this in case the volume of the pipette is large, but it is not necessary in every case.

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## ON THE ACTION OF COLD, CONCENTRATED SULPHURIC ACID, ON LEAD AND ITS ALLOYS.

By LUCIUS PITKIN.

Until quite recently it has been regarded as almost indisputable that the purer the lead, the less action would sulphuric acid have upon it. In opposition to this idea, a very interesting paper was



presented by Mr. James Napier, before the Glasgow Philosophical Society, a full report of which can be found in the *Chemical News* for December, 1880.

Briefly abstracted it is as follows: Sulphuric acid was shipped in cases of sheet lead, all of which either bulged badly or burst. To ascertain the cause of this action, the acid, the lead, and the gas causing the pressure were analyzed.

The acid was of Sp. Gr. 1.842 and the following composition,  $\text{H}_2\text{SO}_4$  99.78— $\text{SO}_2$  0.02— $\text{Pb SO}_4$  0.13— $\text{Ca SO}_4$  0.07.

The lead was of extraordinary purity, containing according to the analysis  $\text{Pb}$  99.96— $\text{Cu}$  0.04. The gas evolved was pure hydrogen.

Exposing a known surface of the lead to the action of cold concentrated sulphuric acid, gas was given off equivalent to 41 cubic inches per square foot lead exposed.

Another sample from a concentrating pan, (No. 1) of the same composition gave under similar circumstances, 16 cubic inches per square foot. A second sample of lead (No. 2) having a composition of  $\text{Pb}$  99.50  $\text{Cu}$  0.08.  $\text{Sb}$  0.42 yielded only  $\frac{1}{8}$  cubic inches per square foot.

As a basis for further experiments, Mr. Napier took a soft lead not analyzed, similar to No. 1, which averaging several determinations yielded 9.4 cubic inches per square foot. Calling this lead No. 3, the following alloys were made and yielded the following amounts of gas by the action of sulphuric acid.

I.	Lead No. 3, 99.25	}	0.25 cu. inch.
	Sb.		
II.	Lead No. 3, 89.88	}	0.10 cu. inch.
	Cu. 0.39		
	Sb. 0.75		
III.	Lead No. 3, 99.63	}	1.42 cu. inch.
	Cu. 0.37		
IV.	Lead No. 3, 99.64	}	2 cu. inch.
	Zn. .37		

The paper was discussed by the society, and the President in summing up, said the following points appeared proven:

1. Chemically pure lead was unsuitable for sulphuric acid evaporating pans.

2. Lead containing certain impurities, and especially zinc, was unsuitable.

3. Antimony seemed to render the lead more durable.



## 4. The subject required further investigation.

It is to this investigation that the remainder of this paper will be devoted.

The lead taken as a basis for the alloys which I have experimented upon, was a chemically pure lead made by Merck, of Darmstadt, and guaranteed by him. The method employed differed from that made use of by Napier, who measured the gas evolved from a known surface of lead.

In the following experiments, the action of the sulphuric acid was measured by the amount of lead or alloy converted into sulphate, which was ascertained by weighing the alloy before immersing in sulphuric acid, and after the action, cleansing from any adhering sulphate and reweighing.

In all forty (40) samples of lead and alloys of known composition were acted upon by the acid and the action measured. In some cases the results may appear anomalous, but not more so than the case reported by Napier, in which lead of the same composition gave off under similar circumstances, in one case 41 cubic inches per square foot, in the other only 16 cubic inches. In the making of the alloys, great care was taken to obtain as homogeneous a mixture as possible, and in order to avoid oxidation, the fusion was performed under a layer of powdered charcoal. The making of 40 alloys was thus by far the most tedious part of the investigation.

The alloys experimented upon were those of lead with antimony, tin, bismuth, cadmium, silver and zinc. After the preparations of the alloys, they were carefully rolled to about the same thickness, and the same surface exposed in each case to the action of the same amount of acid for a like time.

The surface exposed was 2 sq. in., and the amount of acid used 10 c.c. The action was allowed to proceed 24 hours at a temperature of 20° C.

The acid employed was C. P. sulphuric acid of Sp. Gr. 1,825. In the tables the first column gives composition of alloys; the second, the loss of lead per sq. foot of surface exposed, the weight being in grammes; the third, the amount of gas evolved calculated from the quantity of lead converted into the sulphate.

1	C. P. Lead.	1.296 grms.	9 cu. in.
2	"	2.088 "	14.5 "
3	"	2.952 "	20.5 "
4	"	2.232 "	15.5 "

Average loss for pure lead, 2.160 grms. per sq. foot.

Average gas evolved from sq. ft., 15 cu. in.

In all cases quite a vigorous evolution of hydrogen took place at the instant of immersion, while in an hour scarcely any action was perceptible. It will be noticed that the quantity of hydrogen evolved agrees quite closely with the amount given off by lead not in Mr. Napier's experiments.

In the case of the alloys, however, I did not find that the addition of foreign metals produced such a change in the amount of lead converted into sulphate, as the following figures will show.

In computing the amount of gas, the loss is calculated for convenience as entirely lead.

#### ANTIMONY ALLOYS.

5	Pb.	100	Sb.	0.5	parts	1.872	gms.	13	cu. in.
6	Pb.	100	Sb.	1	"	2.016	"	14	"
7	Pb.	100	Sb.	2	"	2.016	"	14	"
8	Pb.	100	Sb.	3	"	1.512	"	10	"
9	Pb.	100	Sb.	5	"	1.584	"	11	"
10	Pb.	100	Sb.	10	"	1.584	"	11	"

It will be seen from this that under the conditions of the experiment, the antimony did not seem to affect the lead to such a degree as in Mr. Napier's researches, although retarding the action of the acid.

It shows, however, what a large amount of antimony may be present without affecting the solubility of the lead.

#### TIN ALLOYS.

11	Pb.	100	Sn.	0.5	parts	2.802	gms.	19	cu. in.
12	Pb.	100	Sn.	1	"	3.744	"	26	"
13	Pb.	100	Sn.	2	"	3.080	"	22	"
14	Pb.	100	Sn.	3	"	2.952	"	21	"
15	Pb.	100	Sn.	5	"	3.232	"	23	"
16	Pb.	100	Sn.	10	"	2.380	"	17	"

In the case of the alloys with tin, the action is in all cases augmented, but does not seem to increase in proportion to the amount of tin present.

#### BISMUTH ALLOYS.

17	Pb.	100	Bi.	0.5	parts	1.800	gms.	12	cu. in.
18	Pb.	100	Bi.	1	"	4.032	"	28	"
19	Pb.	100	Bi.	2	"	1.656	"	11	"
20	Pb.	100	Bi.	3	"	1.728	"	12	"
21	Pb.	100	Bi.	5	"	2.232	"	16	"
22	Pb.	100	Bi.	10	"	3.600	"	25	"

The figures in number 18 are evidently anomalous, and probably were the result of an imperfect admixture or separation of the Bi and Pb. If they are disregarded we would have the general action of bismuth in the alloys with lead as retarding in quantities less than 5 per cent., and above that figure hastening the formation of lead sulphate.

## CADMIUM ALLOYS.

23	Pb. 100	Cd. 0.5	parts	1.728	gms.	12	cu. in.
24	Pb. 100	Cd. 1	"	1.656	"	11	"
25	Pb. 100	Cd. 2	"	1.296	"	9	"
26	Pb. 100	Cd. 3	"	1.728	"	12	"
27	Pb. 100	Cd. 5	"	1.296	"	9	"
28	Pb. 100	Cd. 10	"	3.528	"	24	"

In regard to cadmium we have it decreasing the solubility of lead to a greater extent even than antimony, while above 5 per cent. it raises its solubility.

## SILVER ALLOYS.

29	Pb. 100	Ag. 0.5	parts	1.584	gms.	11	cu. in.
30	Pb. 100	Ag. 1	"	1.728	"	12	"
31	Pb. 100	Ag. 2	"	1.944	"	13	"
32	Pb. 100	Ag. 3	"	1.584	"	11	"
33	Pb. 100	Ag. 5	"	2.016	"	14	"
34	Pb. 100	Ag. 10	"	2.448	"	17	"

Silver seems to exert very little influence, in small proportion, slightly decreasing the action, in large proportion slightly increasing the solubility.

## ZINC ALLOYS.

35	Pb. 100	Zn 0.5	parts	2.664	gms.	18	cu. in.
36	Pb. 100	Zn 1	"	2.304	"	16	"
37	Pb. 100	Zn 2	"	3.816	"	26	"
38	Pb. 100	Zn 3	"	2.664	"	18	"
39	Pb. 100	Zn 5	"	4.032	"	28	"
40	Pb. 100	Zn 10	"	4.392	"	30	"

The solubilities of the alloys of lead and zinc are thus greater than those of lead with any other metal experimented upon. To sum up the results of the work, it appears :

1. The metals, Antimony, Bismuth, Cadmium, and Silver in small quantities, protect lead from the action of the cold sulphuric

acid; while in proportions above 5 per cent., they all, with the exception of antimony, increase the solubility.

2. Antimony when present even to the amount of 10 per cent. decreases the solubility of the lead.

3. Tin and zinc alloys are more affected than pure lead.

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## ACTION OF CONCENTRATED SULPHURIC ACID, AT 100° C., ON LEAD AND ITS ALLOYS.

BY L. PITKIN.

The only work of any importance done, in the estimation of the effect produced upon lead by hot concentrated sulphuric acid, is that of Bauer. The acid used by him was 170° T. (Sp. Gr. 1.848), the amount of lead or alloy taken 0.2 gramme, and the amount of acid used 50 c.c. A brief abstract of his work, so far as it relates to alloys used by me, is here given.

I. *Pure lead*.—The first sensible evolution of gas was at 175° C., a stronger action taking place at 190° C., while at 230°—240° C. all of the lead was suddenly changed to sulphate.

II. *Lead and bismuth alloys*.—

(a). Pb. 90 per cent., Bi. 10 per cent.

Action begins at 150° C., continues quietly to 190° C., when all of the metal is decomposed.

(b). Pb. 96 per cent., Bi. 4 per cent.

This alloy decomposes more quickly than (a), the action terminating at 130°—140° C.

(c). Pb. 99.27 per cent., Bi. 0.73 per cent.

Rapid and sudden decomposition at 160° C.

III. *Lead and antimony alloys*.—

(a). Pb. 90 per cent., Sb. 10 per cent.

A slow and even decomposition takes place, beginning at 190° C., terminating at 240° C.

(b). Pb. 95 per cent., Sb. 5 per cent.

Decomposition begins at 180° C., terminating at 225° C.

(c). Pb. 99 per cent. Sb. 1 per cent.

Action begins at 250°, ends at 280° C.

IV. *Lead and tin alloys*.—Sudden decomposition at 200° C.

The alloys used by me in determining the effect of hot acid were

the same as those employed in estimating the action of cold acid, namely, lead with antimony, tin, bismuth, cadmium, silver, and zinc. The amount of acid was as before, 10 c.c. and the surface exposed 2 sq. in., but the time of exposure was 1 hour, instead of 24 hours, as in testing with cold acid. The amount of gas given off per square foot was not calculated, as that factor would be essential only in the employment of lead for cases. The amount of lead or alloy converted into sulphate per square foot is given in grammes.

The four samples of pure lead, exposed to the action of concentrated acid at 100° C. for one hour, gave very concordant results, as follows :

41.	Pure lead	.....	1.368 Grammes.
42.	"	.....	1.152 "
43.	"	.....	1.224 "
44.	"	.....	1.080 "

The effect of antimony in composition with lead is shown in the following experiments :

45.	Pb. 100 parts,	Sb. $\frac{1}{2}$ part	....	2.952 G.
46.	"	" 1 "	....	3.672 G.
47.	"	" 2 "	....	3.528 G.
48.	"	" 3 "	....	3.096 G.
49.	"	" 5 "	....	2.736 G.
50.	"	" 10 "	....	2.952 G.

Upon immersing the alloy, very little gas was given off, and for 40 minutes the acid remained clear. It then commenced to cloud, and the alloy taken out at the end of the hour was covered with black slime. It will be seen that at 100° C. the action of antimony is not that of a preservative of the lead, as is the case with cold acid ; while from the experiments of Bauer, quoted above, it seems quite likely that at elevated temperatures the alloy with antimony may be more resisting than pure lead. The relative solubilities of the alloys at ordinary temperatures and at 100° C. are by no means constant, and this forms one of the most interesting features of the investigation ; thus, if at common temperatures the alloys with antimony, are found more insoluble than those with zinc, we cannot predicate the same relation with acid at 100° C. In regard to the action of tin upon lead, as affecting its solubility, the following results were obtained :

51.	Pb. 100 parts,	Sn. $\frac{1}{2}$ part....	1.008 G.
52.	"	" 1 "	.... 0.792 G.
53.	"	" 2 "	.... 0.864 G.
54.	"	" 3 "	.... 0.792 G.
55.	"	" 5 "	.... 0.864 G.
56.	"	" 10 "	.... 0.864 G.

It will be remembered that one of the general results obtained from the experiments with cold acid was, that at ordinary temperatures the alloys of lead and tin were more easily attacked than those with antimony, or pure lead itself, and yet at this temperature we see the case reversed.

It is, however, in regard to bismuth that the most curious effects were found to be produced by the composition of the alloy. The following figures will fully explain the peculiar action of the bismuth :

57.	Pb. 100 parts,	Bi. $\frac{1}{2}$ part.....	24.840 G.
58.	"	" 1 "	.... 22.248 G.
59.	"	" 2 "	.... 1.800 G.
60.	"	" 3 "	.... 1.008 G.
61.	"	" 5 "	.... 1.008 G.
62.	"	" 10 "	.... 2.160 G.

The results given in 57 and 58 appear so exceptional, not only in comparison with other alloys, but in regard to the sudden change shown in 59 and 60, that it was decided to make Experiments 57, 58 and 60 in duplicate.

57.	(Duplicate)	Pb. 100 parts,	Bi. $\frac{1}{2}$ ..	25.920
58.	"	"	Bi. 1..	22.750
60.	"	"	Bi. 3..	1.224

We here have a case in which not only the relative solubility in hot and cold acid is changed as regards other alloys, but one in which an excess of the deleterious substance seems to act as a corrective.

The alloys containing  $\frac{1}{2}$  and 1 part of bismuth to 100 of lead gave off gas very plentifully, not only at the start, but throughout the whole hour, while the acid became opaque almost immediately, and the lead sulphate formed could be removed in scales at the end of the experiment.

The experiments with cadmium alloy gave very constant results,

and in general it may be said that, with the exception of bismuth alloy, the figures obtained from the same alloy varied much less than in the corresponding trials with cold acid.

63.	Pb. 100 parts, Cd. $\frac{1}{2}$ part.....	1.440 G.
64.	“ “ 1 “ .....	1.224 G.
65.	“ “ 2 “ .....	1.296 G.
66.	“ “ 3 “ .....	1.080 G.
67.	“ “ 5 “ .....	1.368 G.
68.	“ “ 10 “ .....	1.152 G.

The action of cadmium at this temperature seems to be neither increasing or diminishing the action of the  $H_2SO_4$  on the lead.

In the case of silver combined with the lead, we have the same general behavior, six determinations with varying quantities of silver giving the following results :

69.	Pb. 100 parts, Ag. $\frac{1}{2}$ part.....	1.296 G.
70.	“ “ 1 “ .....	1.080 G.
71.	“ “ 2 “ .....	0.864 G.
72.	“ “ 3 “ .....	0.792 G.
73.	“ “ 5 “ .....	0.936 G.
74.	“ “ 10 “ .....	1.440 G.

The action of zinc in determining the solubility of lead in hot acid is in accordance with its effect on cold concentrated acid—that is, increases the effect of the acid, but the action is not so marked as at ordinary temperatures. The figures for the experiments are :

75.	Pb. 100 parts, Zn. $\frac{1}{2}$ part.....	1.800 G.
76.	“ “ 1 “ .....	1.296 G.
77.	“ “ 2 “ .....	1.152 G.
78.	“ “ 3 “ .....	1.080 G.
79.	“ “ 5 “ .....	1.296 G.
80.	“ “ 10 “ .....	1.080 G.

We can easily see from the results we have obtained the importance of testing the lead employed in  $H_2SO_4$  working, and for this no extended analysis is required. The operation consists simply in immersing the lead in acid, more or less concentrated according to the strength of the acid with which it will be brought into contact in actual working, and at the temperature to which it will be subjected in the manufacture of acid.

Mr. McTear says : “ The simplest safeguard against risk to pans, etc., giving way would be a careful testing of the lead previous to

being made into sheets. For this purpose it will not be necessary to make an analysis, but simply to put clean, thin shavings of lead into a test-tube and cover with pure, cold vitriol, the amount of action would then be clearly visible."

It is, however, clear that the action of cold acid is no sure criterion of the effect that hot acid will have upon the lead; so, to avoid error, it is much safer to test the lead under the conditions of its actual employment.

In order to briefly sum up the results of experiment, it will be advantageous to compare the average of the alloys with pure lead as unity both at ordinary temperatures and at 100° C. The following table will therefore express the average solubility or liability to formation of sulphate of the alloys in terms of lead. In each case the total of the relative solubilities is divided by six (the number of members in the class), for the average solubility of the alloys :

	20° C.	100° C.
Pure lead.....	1.00	1.00
Pb. 100, Sb. 1 to 10 parts.....	0.81	2.75
Pb. 100, Sn. 1 to 10 " .....	1.42	0.75
Pb. 100, Bi. 1 to 10 " .....	1.10	7.69
Pb. 100, Cd. 1 to 10 " .....	0.86	1.10
Pb. 100, Ag. 1 to 10 " .....	0.87	0.93
Pb. 100, Zn. 1 to 10 " .....	1.53	1.10

## OBITUARY.

DR. JOHN LAWRENCE SMITH, died at his home in Louisville, Kentucky, on the 12th of October last, in his 65th year, having been born near Charleston, South Carolina, on the 16th of December, 1818. He had been for some years in rather delicate health and had of late retired very much from the active duties of the laboratory, but maintained his interest in scientific studies.

Dr. Smith pursued his academic studies at the University of Virginia, with which he was later connected as a professor. His medical degree was taken at the Medical College of Charleston. Immediately after this he went to France and Germany, where he zealously followed his medical studies, as also chemistry, physics and mineralogy. While yet a student of medicine at Charleston, he commenced his original work by sending to Silliman's Journal a paper, "On a new method of making permanent magnets by



galvanism." In 1841 he commenced his contributions from Paris in a memoir on the detection of arsenic in the human body, and for the term of his residence abroad he was a regular correspondent of the *American Journal of Science*. The list of his papers in the Royal Society's catalogues embraces seventy-eight titles, down to 1873. In that year Dr. Smith printed in a volume of four hundred pages, 8vo., a collection of the more important of his original researches, embracing forty-seven titles.

Dr. Smith returned to Charleston in 1844 from his first European residence, and commenced his professional work as a physician, which was, however, so little to his taste that he soon abandoned it for the life of an investigator in scientific pursuits. As assayer of the State of South Carolina, he made a study of the soils adapted to the growth of cotton—analyzing, also, the marls used as fertilizers. The Turkish Government soon called him to Constantinople as one of a commission to promote the growth of cotton in Asia Minor. But his active mind developed new lines of investigation and opened up sources of wealth before unknown in the Sultan's dominions, especially in the important discovery of Emery, of which he gives us the first account, setting forth its geology and mineralogy. The Morse telegraph was developed while Dr. Smith was in Turkey, and he wrote immediately to the writer to send him an outfit of telegraphic apparatus which he set up at the Sultan's palace, communicating with the port.

His memoir on the Emery was communicated to the French Academy of Science, and was with distinguished encomiums ordered for publication in the *Memoirs des Savants Etrangers*." He subsequently extended his researches to the Emery of Chester, Massachusetts.

During his brief occupancy of the Chair of Chemistry, at the University of Virginia, he carried out his elaborate research on American minerals, devising and perfecting his method for the determination of alkalies in the silicate. Professor Johnson, in the American edition of Fresenius, says Professor Smith's method is by far the most convenient and accurate for separating alkalies from a silicate, and is universally applicable, except, perhaps, in presence of boracic acid.

In 1854 Dr. Smith was called to Louisville on the resignation of Professor Silliman of the Chemical Chair in the Medical Department, as his successor, and this chair he held for several years. Here he married Miss Guthrie, who survives him. They had no

children. By their marriage Dr. Smith became possessed of an ample fortune which enabled him to devote himself with renewed zeal to scientific work, visiting Europe at frequent intervals to confer with his scientific friends at the French capital, and gather objects of interest, especially in the department of Meteors, to the study of which, as is well known, he devoted much time and contributed many important memoirs. The subject of his last paper, in June of 1883, was "On the Concretions in Meteoric Iron," detailing new methods of research, of which he says, "if my health permits I shall complete them before many months." These were his last published words! Fortunately for science his very large and fine collection of Meteorites was purchased by Harvard University only a few days before his death.

Few have done so much for American mineralogy as Dr. Smith, and done it so well. His works were widely recognized, and few Americans have been rewarded by so many elections to learned societies. The following list embrace the more important both domestic and foreign :

Member of the American National Academy of Sciences ; of the Chemical Society of Berlin ; of the Chemical Society of Paris ; of the Chemical Society of London ; of the Societe d'Encouragement pour l'Industrie Nationale ; of the Imperial Mineralogical Society of St. Petersburg ; Corresponding Member of the Boston Society of Natural History ; of the American Academy of Arts and Sciences ; of the American Philosophical Society ; American Bureau of Mines ; the Societe des Sciences et des Arts de Hainaut, etc. Chevalier de la Legion d'Honneur ; Member of the Order of Nichan Iftahar of Turkey ; Member of the Order of Medjidieh of Turkey ; Chevalier of the Imperial Order of St. Stanislas of Russia : correspondent of the French Institute.

All who knew Lawrence Smith loved him. He was one of the most amiable of men and yet a strong character. One who knew him well says justly of him : " Eminent in his profession he was more than eminent in his home. He was a gentleman, truly, but he was a man of affairs, a man of convictions, a man among men, who though absorbed in scientific pursuits, took a sincere and profound interest in public questions and events. He had not an enemy on earth, despite the positivity and transparency of his opinions, and he goes to his last rest leaving the people with whom he was so long identified to mourn the loss of a citizen of whom all were proud and whom everybody loved and honored."

B. SILLIMAN.

## ABSTRACTS.

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Abstracts from *Journal of the London Chemical Society*. By E. WALLER. Ph.D

**Contributions to the Chemistry of Tartaric and Citric Acid.** Compiled from the manuscripts of B. R. GROSJEAN by R. WARINGTON. (XLIII., p. 331). Different specimens of powdered citric acid lost water of crystallization, by drying over oil of vitriol, very unequally. Of these specimens one lost all its water in 15 days, another in 24 days, while another scarcely lost the whole in 60 days. The cause seems to be different conditions prevailing at the time of crystallization.

M. Grosjean's method for determining "precipitable acid in lemon juice, &c., in—neutralization with soda, addition of calcium chloride, and boiling in a salt or glycerine bath. After filtering off the first precipitate, the filtrate and washings are neutralized with dilute ammonia and concentrated. A second concentration is also made. The precipitates are ignited, and the amount of citric acid calculated from the neutralizing power of the resulting calcium carbonate. The proportion of free acid determined by acidimetry, is usually slightly in excess of the amount of precipitable acid in lemon juice, bergamot juice and lime juice, the ratio being 91 to 103 of precipitable to 100 of free acid. In orange juice it is about 19 per cent. precipitable to 100 of free. Specific gravity is not a safe guide for the value of lemon juice.

The prolonged concentration of tartaric acid per steam causes a partial conversion of the acid into meta tartaric acid. By diluting and boiling the solutions, or allowing them to stand for some time in the cold, the reverse change takes place.

The presence of free sulphuric acid, diminishes very much the solubility of tartaric acid in water.

A hot saturated solution of tartaric acid deposited 50 per cent. of its acid on standing in the cold for some days. With the addition of 100 volumes of brown oil of vitriol (Sp. gr. 1,713) 70 per cent. of the tartaric acid present was deposited.

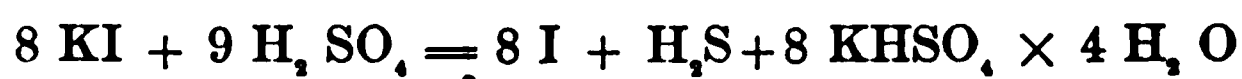
**Note on a Basic Ammonio Copper Sulphate.** S. U. PICKERING. (Vol. XLIII, p. 336).

On adding ammonia to a nearly saturated solution of copper sulphate until the composition was about  $\text{Cu SO}_4, 3 \text{NH}_3$ , and allowing it to stand, a violet blue deposit separated probably consisting of  $\text{Cu SO}_4, 4 \text{NH}_3$ , with some neutral sulphates. On diluting this solu-

tion, a dark violet blue compound,  $\text{Cu SO}_4 \cdot 3 \text{ Cu O} \cdot 2 \text{ NH}_3 \cdot 5 \text{ H}_2\text{O}$ , separated.

A table of the series of analyses made on this precipitate, is given.

**Note on the action of Sulphuric Acid (Sp. Gr. 1.84) upon Potassium Iodide.** H. JACKSON. (Vol. XLIII., p. 339). It was found that when the sulphuric acid was present in large excess the reaction  $2 \text{ KI} + 3 \text{ H}_2 \text{ SO}_4 = 2 \text{ I} + 2 \text{ KHSO}_4 + 2 \text{ H}_2\text{O}$  occurred, whereas when the sulphuric acid was only just sufficient to neutralize the potassium, it was :



I. **Laboratory Notes.** J. H. GLADSTONE and A. TRIBE. (Vol. XLIII., p. 341.)

I. **On the action of Light and Heat on Cane and Invert Sugars.** An attempt to obtain alcohol and carbonic anhydride from sugar solutions by the action of the copper zinc couple, though apparently successful, by close investigation was found to have been altogether unsuccessful. The  $\text{CO}_2$  obtained (at  $100^\circ \text{C}$ ) came from the splitting up of an oxy-carbonate of zinc, while no alcohol was formed, though a substance distilling over and affording the iodoform test, appeared. Heating alone caused a gradual darkening of the sugar solutions, with formation of this substance, together with glucose and some substance acid to test paper. Light appeared from the experiments made to be detrimental to fungoid growths in cane sugar solutions.

II. **Hydroxylamine.** The color produced with this substance and the Nessler reagent resembles that with ammonia when the solution is very dilute. More concentrated solutions give darker colors and precipitates. The hydroxylamine is rapidly converted into ammonia by the zinc copper couple.

III. **Recovery of Iodine from Organic Iodide Residues.** By use of the zinc copper couple the iodine is converted into zinc iodide from which the iodine may be separated by the use of bleaching powder.

IV. **A Residual Phenomenon of the Electrolysis of Oil of Vitriol.** After electrolyzing oil of vitriol it was observed that oxygen gas was evolved from the electrodes long after the current had been broken. It was found to be due to the presence of persulphuric acid.

v. **On alleged tests for Alcohol.** The iodoform test was found to be fallacious. Also the test described by Davy with molybdic acid in sulphuric acid. The blue color may be obtained by several other reducing agents.

vi. **Reaction of the Copper Zinc Couple on Nitric Oxide.** Ammonia was the result of the reaction when the gas was passed over a moistened zinc copper couple. Negative results so far were obtained with free nitrogen.

vii. **On the reducing action of Spongy Lead.** Lead precipitated by zinc from the acetate containing presumably no hydrogen rapidly reduced potassium nitrate to the nitrate. The action was accelerated by heat, and retarded by the presence of sulphuric acid. The lead had no action on solution of potassium chlorate except in the presence of 1 per cent. of  $H_2SO_4$  when it slowly changed to chloride.

**The action of Nitrous Anhydride on Glycerol.** O. MASSON. (Vol. XLIII., p. 348.) On passing nitrous anhydride into glycerol which is kept cold, the liquid increases largely in bulk, and divides into two layers. The smaller layer consists of an aqueous solution of nitrous acid, and various oxidation products of the glycerol. The other layer was found to be impure glyceryl tri-nitrite.  $[C, H, (NO_2)_3]$ . It was purified by repeated distillation over a water bath in a current of hydrogen. It is a mobile volatile liquid of gr. 1.291 distilling at about  $150^\circ C$ . It burns with whitish flame but does not explode under the hammer. It is decomposed by water, nitric oxide gas being evolved. The experimenter was unable to keep the liquid long without decomposition, as it was so very unstable.

**On the preparation of the Pentathionates.** S. SHAW. (Vol XLIII., p. 351). After obtaining the Wackenroder solution by passing  $H_2S$  and  $SO_2$ —(the latter slightly in excess) for 32 hours through distilled water, the solution was neutralized with normal caustic potash, and after concentrating, the solution was placed in partial vacuum over sulphuric acid. The successive crops of crystals contained :—

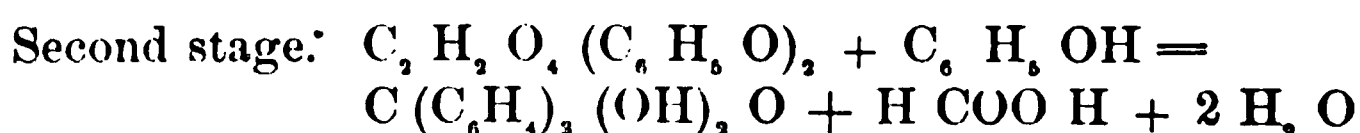
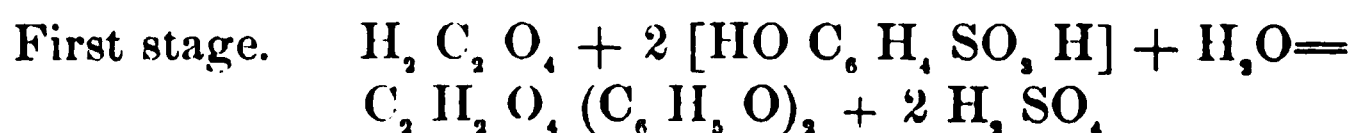
1st Crop	K:S = 2:4	
2d "	K:S = 2:4	
3d "	K:S = 2:4.6	} Mixed.
4th "	K:S = 2:4.6	
5th "	K:S = 2:4.6	
6th "	K:S = 2:5	Selected.

The first crystals gave clear solutions not rendered turbid by addition of Na HO. The last crops of crystals dissolved clear, but the solution become turbid from separation of sulphur on addition of Na HO. A crystallographic description of the crystals is given.

**Note on the action of Allylic Iodide upon Phenol in the presence of Lime or Aluminium Foil.** P. F. FRANKLAND and F. TURNER. (Vol. XLIII., p. 354). Propyl phenol was the result of the reaction with separation of hydriodic acid and free iodine. The probability seems to be that allyl phenol is first formed, which is converted into propyl phenol by the action of the hydriodic acid.

**Note on Pentathionic Acid in connection with the foregoing paper.** W. SMITH. (Vol. XLIII., p. 355). The peculiar fact that the Wackenroder solution when freshly prepared is precipitated by both acids and alkalies, sulphur separating in each case, but after neutralization does not present this phenomenon, being precipitated by acids but not by alkalies, is explained by the fact that at first the free sulphurous acid is present, affording the necessary condition for the formation of a clear solution of thio-sulphate. After evaporation the sulphuric acid is driven off, and the pentathionates left give precipitates with both alkalies and acids.

**On a by-product in the manufacture of Aurin.** A. CLAPAREDE and W. SMITH (Vol. XLIII., p. 358). In preparing aurin by heating together phenol, oxalic and sulphuric acids, a white sublimate appears, which is decomposed by water and by alcohol, with liberation of phenol. They were found to be phenol ortho-oxalic ether.  $C_2H_2O_4 + 2 C_6H_5 OH$ . It is suggested that in the formation of aurin after the formation of phenol sulphonic acid, the oxalic acid unites with the phenol to form the above compound while the sulphonic groups are again separated. In the succeeding stage the action of the sulphuric acid eliminates formic acid and water thus :



**On Samarium and its compounds.** P. T. CLEVE. (Vol. XLIII., p. 362). The laborious method of separating the samaria is described in detail. The atomic weight of samarium was determined from the sulphate believed to be  $Sm_2 (SO_4)_3$ . The average

of six experiments gave in round numbers 150. The results of observations of de Boisbaudran and Thalin on the absorption spectrum of this element in combination is also given. The oxide is white, its salts more or less intensely yellow. Analytical results on the chloride, chloroplatinate, platino-cyanate, nitrate, acetate, sulphate, selenite and oxalate are given, as well as of the samarium potassium sulphate  $2 \text{ Sm}_2 (\text{SO}_4)_3 \cdot 9 \text{ K}_2 \text{SO}_4 + 3 \text{ H}_2\text{O}$  and samarium ammonium sulphate  $\text{Sm}_2 (\text{NH}_4)_2 (\text{SO}_4)_4 + 8 \text{ H}_2\text{O}$ . The element appears to be more closely related to didymium than to any other element.

**On the rate of decomposition of Ammonium Nitrate.** By V. H. VELEY. (XLIIL., p. 370.) The results of numerous experiments are given in detail. The conclusions are :

I. The rate of decomposition of ammonium nitrate into nitrous oxide and water is dependent not only upon the mass of salt undergoing decomposition, but also upon the proportion of free nitric acid present.

II. If the reaction of the salt be rendered alkaline at starting, the rate of decomposition gradually increases while the proportion of free acid increases ; a period of maximum velocity is then reached, corresponding to the greatest proportion of free acid ; from this point the rate decreases very slowly, while the proportion of free acid also decreases.

III. An excess of ammonia obtained either by passing in gas, or by the addition of a basic oxide, will completely stop the reaction, even at temperatures  $50\text{--}60^\circ$  above the normal point of its decomposition.

IV. If the reaction of the salt be rendered acid at starting, the rate of decomposition gradually decreases, while the proportion of acid gradually decreases.

V. After heating the salt for about 13—16 hours the rate of change becomes practically constant.

**On Evaporation in Vacuo.** H. McLEOD. (XLIIL., p. 384.) The paper is unintelligible without the accompanying figures. The idea of the apparatus was suggested by Wrights (Chem. News, XLIV., 311.) and the apparatus devised resembles closely that described by Prof. Mallet (ib XLVII., 218, 252) though it was constructed and used before the appearance of the last named paper.

Violent ebullition of the water is one great difficulty in the process. The evaporation is slower than by the ordinary method being at the rate of about 50c.c. in 2 hours. By the use of sulphuric



acid to absorb the evaporated water the operation can be conducted at only a few degrees above freezing.

**On the Specific Gravity of Parraffin, Solid, Fused and in Solution.** G. BEILBY. (XLIII., p. 388). The paraffin used was carefully purified by repeated crystallization from shale naphtha, steaming and filtering through bone black. It fused at 38°. The results were :

Sp. Gr.	Solid at 21°	874.0
	Dissolved at 21°	795.4
	Fused, (calculated to 21°)	795.6

The solvent used was a paraffin oil of Sp. Gr. 885, at 15.5°.

**On Homologous Spectra.** By W. N. HARTLEY. (XLIII., p. 390). The results of an interesting study of the harmonic relations between lines and groups of lines in the spectra of some of elements, magnesium, zinc, cadmium, copper, silver, aluminium, boron and silicon.

**Thioxalic Ether.** H. F. MORLEY. (XLIII., p. 400). By adding mercaptan in small quantities at a time to chloroxalic ether, the mixture being kept cold in ice water, and afterward boiling off the HCl formed, and finally distilling, some chloroxalic ether first comes off at 128°. After that the temperature quickly rises to 210 and above, the distillate containing the thioxalic ether. This is purified by repeated distillation. It is a colorless liquid with a faint odor like that of garlic. Sp. Gr. at 10° 1.1446. Boils at 217° C (Corr). It is decomposed by caustic potash solution affording potassium oxalate, mercaptan and alcohol. A similar decomposition slowly takes place in water alone.

The results of analysis were :

	Found.	Theory for $C_2H_4SO_2$	
C	44.41	44.24	44.44
H	6.28	1.33	6.17
S	19.24		19.75

The combustion was effected with a mixture of copper oxide, and lead chromate. For the S a modification of Plimpton and Graves's method for chlorine, (Jour Lond Chem Soc., XLI., 119) was used. The method of Carius gave only about 0.1 per cent. S.

Ammonia gas converts thioxalic ether into mercaptan and oxamethane.

An attempt to obtain potassium thio ethyl oxalate by the



action of alcoholic potash on this substance in alcoholic solution resulted in the formation of potassium ethyloxalate and mercaptan.

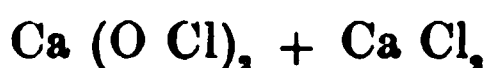
**On the Condensation Products formed by Benzoic Aldehyde with Malonic and Isosuccinic Acids.** Chas. M. STUART. (XLIII., 403.)

Perkin, in investigating the phenomena of condensation occurring between aromatic aldehydes and bodies of the acetic series of acids made use of an anhydride with the sodium salt of the acid, and expressed his belief that the condensation took place between the aldehyde and the anhydride, the sodium salt playing only a secondary part. After a review of the results obtained in regard to this question by Fittig, Payne, Tieman and Claisen, the author gives the results obtained by himself which go to show that the condensation takes place with the sodium salt.

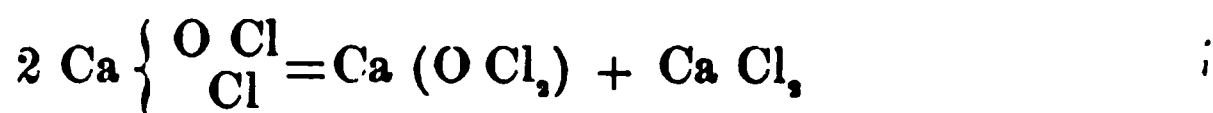
He also finds incidentally that the statement in *Watts' Dictionary* that ethyl cinnamate is scarcely attacked by fuming nitric acid, is erroneous.

**A Contribution to the History of the Constitution of Bleaching Powder.** L. TRAUT O'SHEA. (XLIII. 410.) The methods and results of various experiments are given in detail, The conclusions are :

1. That the excess of hydrate present in bleaching powder, is not a constant quantity.
2. That the formula of the bleaching compound is



3. That by the action of water this compound undergoes the following decomposition :



By *bleaching compound* the author designs to designate the compound at the expense of which oxidizing takes place, as distinguished from *bleaching powder* which is the substance obtained by the action of chlorine on calcium hydrate.

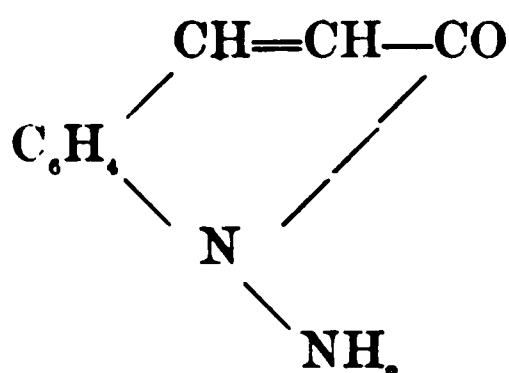
Abstracts from "*Berichte der deutschen chemischen Gesellschaft*," by Percy Neymann, Ph.B.

**On phthalylacetic-acid-ether.** E. Fischer, and H. Koch, (Vol. XVI., p. 651). Phthalylchloride and sodio-acetic-acid-ether act upon each other at ordinary temperature. The reaction is about as follows :



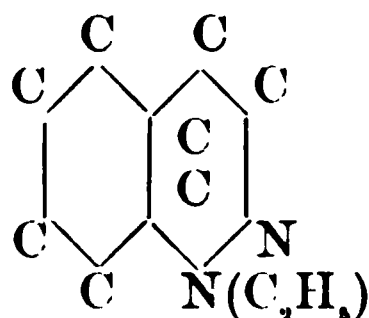
The last compound is called phthalyl-acetic-acid-ether. It is colorless, crystallizes in prisms which have the melting point  $124^\circ \text{C}$ . Succinyl chloride acts in the same manner with sodio-acetic-acid-ether.

**On Chinazol Compounds.** E. Fisher and Hans Kuzel (Vol. XVI., p. 652). Ortho-hydrazin-cinnamic-acid is easily converted into the anhydride corresponding to carbostyryl :



In order to determine if it is possible for a ring connection to exist between carbonyl and the second nitrogen atom of the hydrazinin group, ethylated hydrazinin-cinnamic-acid was examined. It was hoped to obtain this substance by reduction of the nitroso-ethyl-amido-cinnamic acid.

By reducing with zinc dust and acetic acid nitrosamin does not give hydrazin, but is converted into a carbon-acid of the formula  $\text{C}_{10}\text{H}_{11}\text{N}_2-\text{COOH}$  by losing one atom of oxygen. This acid is decomposed by heat into carbonic acid and a base of the formula  $\text{C}_{10}\text{H}_{12}\text{N}_2$ . This has no similarity with hydrazinin, but reminds of chinolin. It probably has the constitution

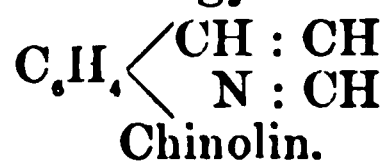


The authors have called it "Ethyl-chinazol," and the corresponding acid "Ethyl-chinazol-carbon-acid."

**On a Reaction of Aldehydes.** F. Penzoldt and Emil Fisher. (Vol. XVI., p. 657). When mixing diabetic urine with an alkaline solution of diazobenzol-sulfonic acid a red color was observed after 10—15 minutes, which gradually assumed a violet color. The same reaction was observed in a solution of pure grape sugar, and investigation showed that this was a reaction common to aldehydes. In the case of stable aromatic aldehydes the reaction only takes place by addition of sodium amalgam.

**On the Compounds of the Hydrazins with Ketones.** H. Reisenegger. (Vol. XVI., p. 661). Phenylhydrazin combines with the various ketones at ordinary temperature in the proportion of equal molecules, by giving off water. The products are constant in the presence of water and alkalies, but are easily reconverted into the generators by acids. The same reaction can be executed with secondary hydrazins.

**On Cinnolin Derivatives.** V. v. Richter. (Vol. XVI., p. 677). The investigation was undertaken with the idea of preparing ortho-oxy-acteo-phenon  $C_6H_4(OH)COCH_3$  from ortho-nitro-phenyl-propionic acid, in order to arrive at the synthesis of alpha-cumaric acid. However, the investigation led to something entirely different, by exhibiting a new group of compounds which are to be regarded as derivatives of a substance  $C_8H_6N_2$ , which, because of its analogy with chinolin the author has termed cinnolin.



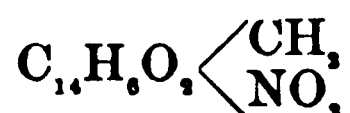
The cinnolin can be regarded as a chinolin in which the CH group tied to oxygen is replaced by a nitrogen atom ; it contains a six-jointed closed ring consisting of four carbon and two nitrogen atoms.

**On Sulphonic Acids of Hydrochinon.** A. Seyda. (Vol. XVI., p. 687). After having prepared hydrochinon by Nietzki's method, the author prepared the hydrochinon-mono-sulphonic acid and hydrochinon-di-sulphonic acid. To obtain the mono-acid one part of hydrochinon is heated with 8 parts of mixed sulphuric acid at 50° C. for three hours with constant stirring. After 24 hours the mono-acid as a rule crystallizes out. The barium, zinc, and potassium salts are easily prepared.

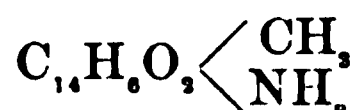
The hydrochinon-di-sulphonic acid is prepared by dissolving one part of hydrochinon in 5 parts of fuming sulphuric acid and heated at 100° to 110° for one hour. The acid gradually separates, and

after cooling a thick, sticky mass is produced. The free acid is obtained by precipitating the barium from the barium salt with sulphuric acid. It crystallizes in long thick needles. It has an astringent taste and effloresces in the open air.

**On Nitro-amido and Oxy-methyl-anthrachinon.** H. Roemer and W. Link. (Vol. XVI., p. 695). For some time past there is found in crude anthracene at times a higher homologue termed methyl-anthracene. It is especially found in the American product and is very stable. Oxidation produces methyl-anthrachinon, and even after sulphurizing and subsequent fusion with caustic soda the methyl group is not eliminated. Nitromethyl-anthrachinon is prepared by dissolving two parts of methyl-anthrachinon in conc. sulphuric acid and adding one part of nitric acid sp. gr. 1.48. The color changes from original red to yellow and a crystalline precipitate gradually deposits. After 24 hours the mass is poured into water, the product crystallizes and is purified from acetic acid. The melting point is at 269° to 270°. the formula is



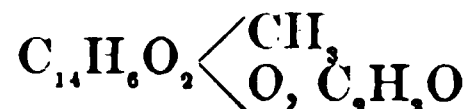
Amido-methyl-anthrachinon is prepared by reducing the nitro compound with alkaline solution of stannous oxide. The amido compound melts at 202°. It is dark red in color and crystallizes in needles. The formula is



Acetyl-amido-methyl-anthrachinon crystallizes in bright red needles, melts at 176° and 177° and has the formula



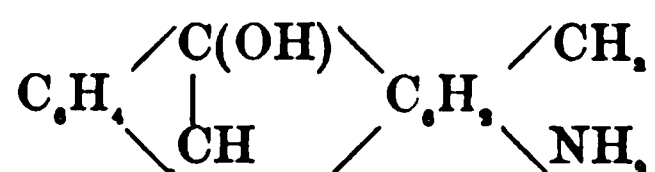
Acetyl-oxymethyl-anthrachinon crystallizes from alcohol in orange colored needles of the melting point of 177°, and the formula is



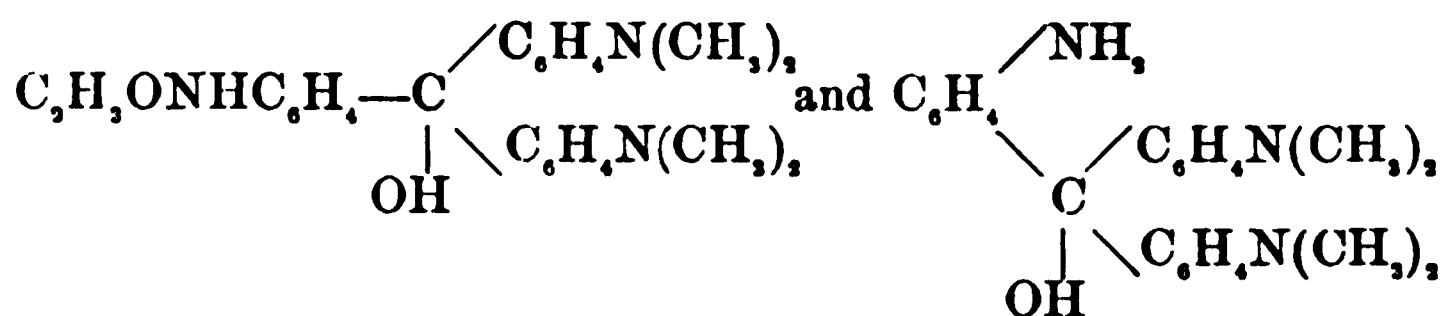
It is remarkable that all these derivatives have approximately the same melting point :

Methyl-anthrachinon	.	.	.	.	177°
Acetyl-amido-methyl-anthrachinon	.	.	.	.	176°-177°
Oxy-methyl-anthrachinon	.	.	.	.	177°-178°
Acetyl-oxy-methyl-anthrachinon	.	.	.	.	177°

**Amidomethylanthrol.** H. ROEMER and W. LINK. (Vol. XVI., p. 703.) If amido-methyl-anthrachinon is heated with hydroiodic acid of sp. gr. 1.7 and red phosphorus to boiling for one hour, then water added and washed until ammonia no longer produces a precipitate in the filtrate very little substance remains on the filter. This compound is difficult to obtain. The reaction is entirely different when hydroiodic acid is used of the sp. gr. 1.96. If amido-methyl-anthrachinon and half of its weight in red phosphorus are heated with this on a water bath for one to two hours very little substance will go into solution. The residue is soluble in dil. hot hydrochloric acid and conc. hydrochloric acid precipitates yellow needles, the hydrochloric acid salt of a base. Washing with water separates the acid and leaves a base, melting at 183° C. The formula, according to analysis, was found to be (C<sub>11</sub>H<sub>11</sub>NO). The compound is called amido-methyl-anthrol



**On the Violet Derivatives of Triphenylmethane.** O. FISCHER and L. GERMAN. (Vol. XVI., p. 706.) The authors assume, contrary to former investigations (Berichte, XII., 800) that the base of methyl violet contains six methyl groups, and they base their opinion on the results obtained by boiling acetyl-tetramethyl-para-rosanilin with conc. hydrochloric acid. Acetyl is separated and the violet obtained is the same as that obtained by direct oxidation of tetramethyl-para-leukanilin with chloranil.



Acetyl-tetramethyl-para-rosanilin (green).

Methyl violet.

**On Tetra-Hydro--Chinolin.** L. HOFFMANN and W. KOENIGS. (Vol. XVI., p. 727.) It is prepared by heating on a water bath a solution of one part of chinolin in about thirty of strong hydrochloric acid and gradually adding three to three-and-a-half parts of granulated tin. The excess of acid is driven off and after addition of concentrated solution of caustic alkali steam is driven through the resulting mass carrying chinolin and tetra-hydro-chinolin with it. To



Abstracts—*Zeitsch. für Anal. Chemie.*, by F. P. Venable, Ph. D.

**Determination of Nitrogen in Organic Bodies.** By J. KJELDAHL. (*Zeitsch. Anal. Chem.*, 22.366). The author proposes to substitute an oxidation, by means of permanganate, of the organic substance in acid solution for the oxidation in alkaline solution as in the process of Wanklyn.

The sample is first strongly heated with sulphuric acid (concentrated). Nearly all substances are thus brought into a condition in which the nitrogen can be completely converted into ammonia. The principle of this conversion is to heat the substance with a sufficiency of sulphuric acid to a temperature near the boiling point of the acid, and then to oxidize the solution thus obtained by means of an excess of dry powdered permanganate. The nitrogen present in the organic compounds is under these circumstances completely converted into ammonium sulphate. After the oxidation and a supersaturation with soda, the liquid can be distilled off and determined according to the usual methods. It is essential for this process that the ammonium sulphate suffer no decomposition by the high temperature and subsequent treatment with potassium permanganate, which is attended with a very violent reaction. Several experiments gave proof that no such decomposition took place.

In carrying out the process, the substance is placed in a 100 c.c. flask with a long neck, and sulphuric acid is added in sufficient excess. It is then heated over a small flame. The liquid is at first black and tarry but finally clear. The heating lasts about two hours. The temperature should be a little below the boiling point. The permanganate is introduced in small portions, the flame being removed. The completion of process is known by the appearance of a green color. When sufficiently cool, water is added. It is introduced into the distilling apparatus, soda solution added (zinc turnings having first been added to prevent bumping), and the ammonia distilled over.

Nitrates in the presence of organic matter are converted into ammonia. The application of the process is doubtful where certain alkaloids and nitrogen in the form of volatile acids occur.

**Estimation of Phosphoric Acid as Magnesian Pyrophosphate.** By DAVID LINDS. (*Chem. News*, 48.217). The author used, in the experiments, microcosmic salt. The precipitates were made direct with magnesia mixture, 6 p.c.  $\text{NH}_3$  added, and the washing done with 1.2 p.c.  $\text{NH}_3$ . The experiments were car-

ried out in sets of eighteen and averages taken. The following conclusions were drawn:

Moderate heating does not influence the results—precipitation at or near the boiling point gives results that are too high.

Large excess of magnesia mixture, if added gradually, does no harm. A large excess must be used in the presence of ammonium chloride, oxalate, or citrate. If added all at once the results are too high. If constantly stirred, 2 c.c. may be added at a time. When neutral magnesia mixture is used (prepared by boiling to expel ammonia) the precipitation is in complete until ammonia has been added. Results then accurate, and precipitate easily washed.

Precipitation in presence of much free ammonia gives a voluminous precipitate which is hard to wash. If ammonium citrate or oxalate is present the precipitate is crystalline and compact. Using a 2.5 p.c.  $\text{NH}_3$  solution, the author finds its solvent action 0.0002 grm. per 100 c.c. used in washing.

Ammonium oxalate, citrate and chloride have a solvent action on the precipitate, and also, apparently, a power of hindering its formation. A large excess of magnesia mixture prevents both of these actions.

These results may be compared with the investigations of Ogilvie (Chem. News, 31.274, 32. s.12.70.).

**Separation of Bismuth from Copper.** By JULIUS LOWE. (Zeit. Anal. Chem. 22.495). Solutions of Bismuth and Copper are precipitated by potassium and sodium hydroxide, and completely redissolved in excess in presence of glycerine. On adding a solution of glucose, and heating, both are precipitated, the copper as cuprous oxide, and the bismuth as finely divided metal. If however, after addition of glucose the solution stands several hours in the cold, only the copper is precipitated (a little of the bismuth is deposited after several days). Quantitative experiments gave fair results.

**Precipitation of Manganese by air loaded with Bromine-vapor.** By WOLF. (Zeit. Anal. Chem., 22.520). Instead of the usual precipitation of manganese with bromine water, after iron has been removed by sodium acetate in iron-ore analyses, the author recommends the use of the corresponding ammonia-salts, and claims to avoid all danger of forming nitrogen bromide, the incomplete precipitation of the manganese, etc., as well as making a more rapid determination, by forcing or drawing



through the precipitation flask a stream of air which has first bubbled through bromine water ; 15 to 20 minutes suffices for complete precipitation even where the proportion of manganese is large.

**Behavior of Silver Chloride, Bromide and Iodine toward Bromide and Iodine.** By PAUL JULIUS. (Zeitsch. Anal. Chem., 22.523). A series of experiments leads the author to believe that any halogen used in excess is capable of expelling any of the others from their silver compounds. Thus the iodide or mixture of silver iodide and bromide can be changed into the bromide if air loaded with vapor of bromine, is forced over them when fused. Silver chloride, too, is changed into bromide after 1-2 hours action of bromine vapor. A much longer time, (6-10 and 3-4 hours respectively), is required to substitute chlorine or bromine in their silver compounds by iodine.

**Test for Gallic Acid.** By SIDNEY YOUNG. (Chem. News 48.31.). When an aqueous solution of gallic acid is treated with a solution of potassium cyanide, a beautiful red coloration is produced, which, however, disappears after a short time, if the liquid is not disturbed, a brownish-yellow color being left. Pure tannic acid gives no coloration with potassium cyanide, but commercial tannic acid invariably contains a certain amount of gallic acid. The reaction is, however, feeble. The reaction serves them to distinguish between the acids, and detect gallic acid in presence of tannic.

**Identification of Minute Quantities of Silver Cyanide.** By C. L. BLOXAM. (Chem. News, 48.59). Precipitated silver cyanide is amorphous under the microscope. Warmed with a drop of ammonia, however, it will form distinct needle-like crystals. Silver chloride treated in the same way gives minute octahedra. The sulphocyanide also forms needles, but its absence can be proved by the ferric chloride test. These needles are obtained also by boiling with a strong solution of sodium carbonate or with dilute nitric acid.

**An Indicator to Show the Neutral End-reaction in Alkalimetry and Acidimetry.** By A. GAWALOVSKI. (Zeitsch. Anal. Chem., 22.397). The usual indicators enable one to determine when either alkali or acid is in slight excess. If one mixes alcoholic solutions of phenolphthalein and dimethyl-aniline orange, (so-called methyl orange), an indicator is obtained which becomes

deep red when alkali is in excess, and is tinged rose color by an excess of acid. If neither is in excess and the neutral-point has been reached, the solution has from the indicator a clear citron-yellow color. The changes of color require 4–5 seconds. The mixed solutions of the indicators remain for at least five days perfectly good.

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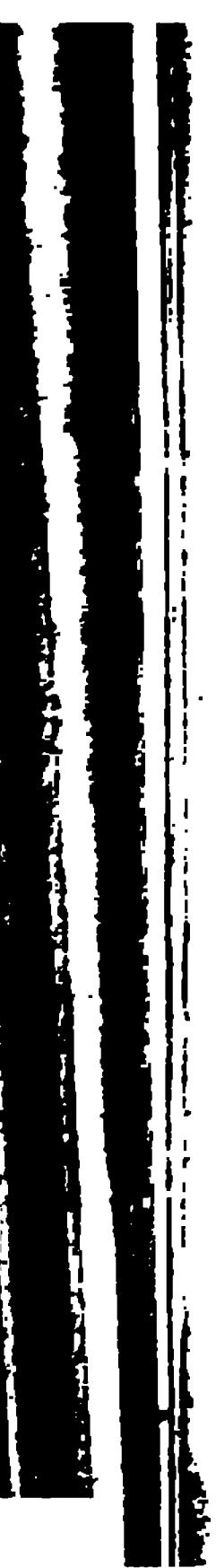
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